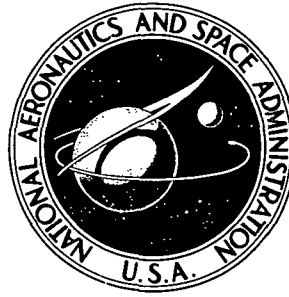


**NASA TECHNICAL
MEMORANDUM**



NASA TM X-2635

NASA TM X-2635

CHEMICAL PROCESSING MANUAL

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TECHNICAL REPORT STANDARD TITLE PAGE

1. REPORT NO. NASA TM X-2635	2. GOVERNMENT ACCESSION NO.	3. RECIPIENT'S CATALOG NO.	
4. TITLE AND SUBTITLE Chemical Processing Manual		5. REPORT DATE November 1972	
		6. PERFORMING ORGANIZATION CODE	
7. AUTHOR(S) F. J. Beyerle		8. PERFORMING ORGANIZATION REPORT # M802	
9. PERFORMING ORGANIZATION NAME AND ADDRESS George C. Marshall Space Flight Center Marshall Space Flight Center, Alabama 35812		10. WORK UNIT NO.	
		11. CONTRACT OR GRANT NO.	
		13. TYPE OF REPORT & PERIOD COVERED Technical Memorandum	
12. SPONSORING AGENCY NAME AND ADDRESS National Aeronautics and Space Administration Washington, D. C. 20546		14. SPONSORING AGENCY CODE	
15. SUPPLEMENTARY NOTES			
16. ABSTRACT <p>This manual presents all documents directly related to chemical processes that were prepared by the Product Engineering and Process Technology Laboratory and are pertinent to the continuing technology of spacecraft development.</p> <p>Chemical processes presented in this document include cleaning, pickling, surface finishes, chemical milling, plating, dry film lubricants, and polishing. All types of chemical processes applicable to aluminum, for example, are to be found in the Aluminum Alloy Section. There is a separate section for each category of metallic alloy plus a section for non-metals, such as plastics. The refractories, super-alloys and titanium, are prime candidates for the Space Shuttle; therefore, the chemical processes applicable to these alloys are contained in individual sections of this manual.</p>			
17. KEY WORDS		18. DISTRIBUTION STATEMENT	
19. SECURITY CLASSIF. (of this report) Unclassified	20. SECURITY CLASSIF. (of this page) Unclassified	21. NO. OF PAGES 918	22. PRICE \$9.00

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ACKNOWLEDGEMENT

The assistance of Theodore W. Lewis and David D. Webb of S&E-PT-MXC and W. L. Hayes, S. A. White, and D. F. Jackson of Hayes International under a support contract in the compilation, editing, and proof-reading of this manual is gratefully acknowledged.

PREFACE

A wide range of chemical processes are required within the Process Engineering Laboratory for the development of space flight vehicles. Modifications are needed within the same chemical processes for different type materials. In order to meet the rigid aerospace requirements of the finished product, close controls have to be maintained on the various operations. These facts point out the need for a wealth of accurate data relative to chemical processes for the aerospace alloys.

As a result of this need many documents have been prepared by the Process Engineering Laboratory establishing criteria for chemical processes such as cleaning, pickling, passivating, descaling, chemical milling, plating and many more. These documents were known as Manufacturing Spec. (MS), Manufacturing Procedures (M-ME-MPROC), and Manufacturing Process (MPD). All are basically manufacturing process data documents.

The need of a comprehensive chemical process manual to provide a ready reference to NASA and industry personnel involved in chemical processing of space hardware was recognized by those associated with the operation.

GLOSSARY

- absorption — penetration of a substance into the body of another; to take in and incorporate; assimilate.
- acid — A substance whose molecules ionize in water solutions to give off hydrogen ions; a substance which registers less than 7 on the pH scale.
- aerosol — finely divided particulates suspended in air.
- adsorption — adhesion of the molecules of a gas, liquid, or dissolved substance to a surface; the taking up of one substance at the surface of another.
- aerobic microorganisms (or obligate aerobic) — grow only in the presence of free or atmospheric oxygen.
- aerosol — a suspension of microscopic solid or liquid particles in air or gas.
- agglomeration — the combining, joining, clumping, or clustering of two or more particles or droplets by any means.
- air-cleanliness class — each class of air cleanliness is determined by the particle count per unit volume, based on tabulation of particles 0.5 micron and larger or 5.0 microns and larger. Examples of standard air-cleanliness are described and illustrated in Paragraph 5.1 and Tables I and II in FED. STD. No. 209a.
- airlock — a chamber with doors functioning to maintain pressure during entry to and exit from an enclosed area.
- air shower — a chamber with interlocked doors and equipped with an exhaust system, having numerous air nozzles arranged in a predetermined pattern, for the purpose of forcibly blowing loose particles, fibers, dust, and other particulate matter from the person and garments.
- alkali — any base or hydroxide that is soluble in water and can neutralize acids; a substance that registers more than 7 on the pH scale.

GLOSSARY (Continued)

ambient condition — environmental conditions such as pressure, temperature, humidity, etc., which are normal for one specific location.

assembly — two or more parts; subassemblies, or any combination thereof, joined together to perform a specific function.

bio — prefix indicating life.

carbohydrate — organic compound composed of carbon, hydrogen, and oxygen; examples: sugars and starches.

catalyst — a substance which changes the rate of a reaction (chemical), usually by increasing it, without itself being consumed.

class 100 clean station — no more than 100 particles over 0.5 micron in size per cubic foot.

cleanable — capable of being cleaned to specified levels without detrimental effect.

cleaning — act of removing unwanted substances.

cleaning agent — any compound or substance which promotes the removal of soils through mechanical or chemical action.

cleanliness level — an established maximum allowable distribution of contamination of a given size and quantity in a stipulated area or volume.

clean packaging — the application of clean preservation and packaging measures and material to maintain the cleanliness of a clean item during handling, storage, or shipment.

clean room — a clean room is an enclosed area employing control over the particulate matter in air with temperature, humidity, and pressure control as required. To meet the requirements of a "clean room", the area must meet the particulate count as specified in FED. STD. 209a, paragraph 5.1.3.

GLOSSARY (Continued)

compound — a substance formed by the chemical union of two or more elements.

contaminant — any material, substance or energy which is unwanted or adversely affects the contaminee.

contaminate — the act of introducing any contaminant; to make impure or unclean; to pollute, defile, sully, taint, or soil.

contamination — the presence of one or more contaminants in or on the contaminee.

contamination control — the planning, organization, and implementation of all activities needed to determine, achieve, and maintain a required cleanliness level in, on, or around the contaminee.

controlled area — any enclosure which has a degree of control of contaminants in air, gases, and fluids (may include temperature, humidity, and pressure) and which will not qualify as a clean room.

decontamination — the process of removing unwanted matter; the reduction of contamination to an acceptable level.

deionization — the removal of mineral ions from water.

design packaging — the enclosure of a unitized assembly of components, or parts in a protective container, covering or coating which is an integral part of the item and will enhance the function of the item; usually involves the use of sealed cans, encapsulating compounds, etc.

distillation — the phase changes from liquid to vapor to liquid; a process for removing impurities from liquids.

electrostatic — pertaining to the phenomena due to attractions and repulsions of electrical charges.

GLOSSARY (Continued)

element — the simplest type of matter; elements cannot be chemically broken down into simpler substances; everything we know is made up of elements; either by themselves or in combination with each other; oxygen is an element, as are hydrogen, iron, carbon, and nitrogen.

emulsion — a liquid in which other particles or liquids are suspended; a characteristic of some cleaning agents in holding and carrying away soils.

entrapment — the act of securing and holding, as dirt is entrapped in a filter.

environment — the total of all factors which might influence or cause contamination of a contaminee. The primary factors of environment are the forms, air, gas, liquid, solid, or surfaces.

environmental package — a clean material closure, wrap or container over the intimate package that is sealed to provide an additional contamination barrier to protect the intimate package and its contents from environmental elements.

filter — a device using a medium to selectively separate certain substances from a given environment.

filtration — the process of removing contaminants from a gas or liquid by passing them through a porous media.

frequency — the number of vibrations, or cycles per second.

fungi — microorganisms which lack chlorophyll and generally possess filamentous vegetative structures, i.e., yeasts, molds, rusts, mildews, smuts, etc.

gas — a state of matter in which the molecules are practically unrestricted by cohesive forces. A gas has neither shape nor volume, and in form is neither liquid nor solid.

GOX — Gaseous oxygen

GLOSSARY (Continued)

gram — the unit of mass in the metric (scientific) system.

hertz (Hz) — one cycle per second.

horizontal laminar airflow clean room — a room equipped with one entire vertical wall of HEPA filters, through which the air passes at a predetermined speed to an exhaust wall directly opposite the HEPA filter wall. The entire body of air moves horizontally across the room along essentially parallel flow lines at uniform velocity.

hydration — combination with water.

hydrocarbon — a chemically identifiable compound of carbon and hydrogen.

intimate cushioning — a clean material used to protect additional packaging materials from puncture or damage caused by the clean item.

iodophore — organic compounds of iodine in which iodine is loosely combined with some surface active agent; nonstaining, nonirritating, and practically odorless.

kilocycle (kc) — 1000 cycles

kilohertz (kHz) — 1000 Hertz or 1000 cycles per second.

latent heat — the calories of heat required to change one gram of liquid to vapor at the boiling point.

liter — metric unit of capacity equal to the volume of one kilogram of water of 4°C and at standard atmospheric pressure.

liquid — a state of matter in which the molecules are relatively free to change their positions with respect to each other but restricted by cohesive forces so as to maintain a relatively fixed volume.

GLOSSARY (Continued)

LOX — liquid oxygen

macro — prefix meaning large, usually visible without the aid of a microscope.

micro — prefix meaning small.

micron — a unit of measurement equal to one-millionth of a meter or approximately 0.00003937 inch (e.g., 25 microns are approximately 0.001 inch).

millimicron — one thousandth of a micron, abbreviated mu.

milliliter — one thousandth of a liter, abbreviated ml.

molecule — the smallest unit of a compound.

molecular attraction — the adhesive forces exerted between the surface molecules of two bodies in contact.

nylon — a polymide with high abrasion resistance, but relatively poor moisture barrier characteristics.

organic — designating any chemical compound containing carbon.

oxide — a binary compound of oxygen with some other element or with a radical.

packaging — the application or use of appropriate closures, wrappings, cushioning, containers, and complete identification, up to, but not including the shipping container and associated packing.

particle — a piece of matter with observable length, width, and thickness; usually measured in microns.

particulate matter — the general term applied to matter of miniature size, with observable length, width, and thickness, and contrasted to nonparticulate matter without definite dimension.

GLOSSARY (Continued)

- pH — potential of hydrogen; a symbol for the logarithm of the reciprocal of the hydrogen ion concentrations, expressed in gram atoms per liter of a solution; used to indicate acidity or alkalinity.
- plating — cultivation of microorganisms in Petri dishes containing a solid nutrient medium.
- polyethylene — a thermoplastic polymer capable of being produced in thin sheets; exhibits good moisture-vapor barrier qualities but has a high sloughing tendency.
- precleaning — the cleaning which is accomplished outside of a clean area, for the purpose of removing contaminants such as rust, oxidation, grease, oil, heavy scale or soil deposits to control the amount of contaminants brought into the clean room.
- rad — amount of ionizing radiation producing an absorbed dose of 100 ergs per gram in some specified tissue material.
- radiation — the process of emitting radiant energy in the form of waves or particles.
- reagent — a substance used to produce a characteristic reaction in chemical analysis.
- solid — a state of matter in which the relative motion of molecules is restricted; the molecules tend to retain a fixed position relative to each other.
- solvent — that solution or constituent of a solution which exhibits the capability to dissolve other substances through chemical action.
- specification — a document which establishes specific and individual requirements for processes, product, and product performance.
- specific heat — the thermal capacity of a liquid as a ratio of that of water at 15° C.

GLOSSARY (Concluded)

standard — a uniform method of defining basic parameters for processes, product, or measurements.

surface tension — force by which the surface film of all liquids tends to contract.

toxic — poisonous

transducer — a device that converts energy from one form to another. An ultrasonic transducer changes high-frequency electrical energy into high-frequency sound waves.

ultrasonics — the physical science of those acoustic waves that oscillate in the approximate range of 18 to 80 kHz.

vapor — the gaseous form of any substance which is usually a liquid or a solid.

vertical laminar airflow room — a room equipped with a ceiling of HEPA filters, with a grated or perforated floor for the exhausting of air issuing from the ceiling filters; the airflow is vertical, and moves within the walled area along essentially parallel lines at uniform velocity.

wavelength — the distance between two undulations of a wave train.

CHEMICAL PROCESSING MANUAL

SECTION I. INTRODUCTION

This manual contains operational procedures for all chemical processes currently in use in the Process Engineering Laboratory. The basic operations include cleaning, pickling, surface finishes, chemical milling, plating, dry film lubricants, and polishing. Sufficient details of the operations are included so the operators may use them for reference.

The basic operations are applicable to a wide variety of metallic and non-metallic materials. In most cases the procedure has to be modified for a specific type material. For this reason the chemical processes are indexed under the material rather than the process. This method of indexing affords expeditors, planners, etc., a quick summary of all the chemical processes applicable to a specific material or product.

SECTION II. CHEMICAL PROCESSES

For the purpose of convenience, a general description of chemical processes is given prior to specific treatments of materials. These processes consist primarily of general cleaning methods of metals and is intended to be used as a guideline only. These procedures have been reduced from standard metals handbook¹ and are applicable to chemical processes related to aerospace work.

2.1 CLEANING -- GENERAL

Cleanliness is the most important facet of chemical processing. All related processes and surface treatments depend to a large extent on the cleanliness of the material to be processed.

There are various types of cleaning processes now in use. These include acid cleaning, alkaline cleaning, solvent cleaning, emulsion cleaning, electrolytic cleaning, pickling, and descaling. All of these cleaning methods, either alone or in conjunction with others, are employed in the cleaning of specific materials.

Selection of a cleaning process is influenced mainly by the material to be cleaned, type of soil to be removed, the required degree of cleanliness, and the cost. Except for scale-removal processes, the size and shape of workpieces seldom influence the type of cleaner used but may determine the method of cleaning, such as immersion or spray, and the handling techniques that are employed.

Types of soil may be broadly classified into six groups: (1) pigmented drawing compounds, (2) unpigmented oil and grease, (3) chips and cutting fluids, (4) polishing and buffing compounds, (5) rust and scale, and (6) miscellaneous surface contaminants, such as compounds and residue from magnetic particle inspection.

The cleaning process is generally performed in two phases, precleaning and final cleaning. Depending on the type and amount of soil involved, the precleaning step can often be omitted.

1. See Section XVI, References and Specifications.

2.2 PRECLEANING

The purpose of precleaning is to remove as much of the soil as possible or condition it for complete removal in the final cleaning process. Precleaning also affords less chance of the final cleaning bath to become contaminated.

2.2.1 Solvent Cleaning

Solvent cleaning is most widely used as a preliminary or conditioning cleaner to decrease the time required in, and contamination of, the final cleaner. It is performed in a soak, hand, or spray operation using petroleum or chlorinated solvents and functions by dissolving all or part of the soil. Vapor degreasing has proved especially effective for removing soluble soil from crevices, such as rolled or welded seams that may permanently entrap other cleaners. Vapor degreasing is particularly well adapted to cleaning oil-impregnated parts and for removing solvent-soluble soils from the interior of storage tanks.

Solvent cleaning may be used to remove the common oils and greases from metal parts. Methods vary from static immersion to the use of a multistage washer. Seven methods of solvent cleaning, listed in order of their ascending effectiveness, are as follows:

1. Static immersion
2. Immersion with agitation of parts
3. Immersion with agitation of both the solvent and parts
4. Immersion with scrubbing
5. Pressure spraying in a spray booth
6. Immersion scrubbing, followed by spraying
7. Multistage washer

2.2.2 Emulsion Cleaning

Emulsion cleaning is a process for removing soils from the surfaces of metal by the use of common organic solvents dispersed in an aqueous medium with the aid of an emulsifying agent. Depending on the solvent used, cleaning is done at temperatures from room temperature to 140 - 180° F.

An emulsion system must contain two liquids, mutually insoluble or nearly so, one of which usually is dispersed in the other in the form of globules. In general, one of these liquids is a hydro-carbon and the other is water. The dispersed phase is that which is distributed as globules in the other liquid, known as the continuous phase. Because oil and water do not mix, an oil-in-water dispersion that does not contain an emulsifying agent (or dispersant) requires constant mechanical agitation to prevent immediate separation of oil and water into two layers.

The stability of emulsion cleaners depends on the properties of emulsifying agents that are capable of causing oil and water to mix and form a more stable emulsion. These emulsifying agents are of two general types: Those that promote the formation of oil-in-water emulsions (water constituting the continuous phase) and those which form water-in-oil emulsions (in which water is the dispersed phase).

Emulsion cleaners are broadly classified into four groups, on the basis of stability; there is some overlapping of definitions, because stability is a relative term.

A "stable", "stable single phase", or "permanent" emulsion is one in which the discontinuous phase is dispersed throughout the continuous phase, and which requires no more agitation to maintain a uniform dispersion than that provided by thermal gradients and the motion of the work being cleaned.

In common with a stable emulsion, an "unstable" or "unstable single phase" emulsion has a uniformly dispersed discontinuous phase, but requires moderate to considerable agitation to maintain dispersion.

The solvent is generally of petroleum origin and may be paraffinic, naphthenic, or aromatic. Solvents are available with boiling points of 140 to 500° F and flash points ranging from room temperature to above 200° F. The lower-boiling solvents are usually more effective in removing soils; however, fire hazards and evaporation losses increase as boiling and flash points decrease.

Emulsifiers include polyethers and high molecular-weight sodium or amine soaps of hydrocarbon sulfonates, amine salts of alkyl aryl sulfonates, fatty-acid esters of polyglycerides, glycerols, or polyalcohols. Cationic agents also are used in formulating emulsions.

Emulsifiers must have some solubility in the oil phase. When solubility is low, it can be increased by adding a blending agent, such as a higher-molecular-weight alcohol, ester, or ether. These additives are soluble in oil and water.

2.2.3 Mechanical Cleaning

Abrasive blast cleaning is widely used for removing all classes of scale and rust from mill products, forgings, castings, weldments, and heat treated parts. Depending on the finish requirement, blasting may be the sole means of scale removal or it may be used to remove the major portion of scale, with pickling employed to remove the remainder.

Tumbling is often the least expensive process for removing rust and scale from mill products, forgings, castings, weldments, and heat treated parts. Part configuration and size are the primary limitations of the process. Tumbling in dry abrasives (deburring compounds) is effective for removing rust and scale from small parts of simple shape. However, parts of complex shape, with deep recesses and other irregularities, cannot be descaled uniformly by tumbling and may require several hours of tumbling if that method is used. The addition of descaling compounds instead of deburring compounds will often decrease the required tumbling time by 75 percent.

Brushing is the least used method of descaling parts, although it is satisfactory for removing light rust or loosely adhering scale. It is better suited for work pieces formed from tubing than for castings or forgings.

2.2.4 Acid Cleaning

Acid cleaning is a process in which a solution of a mineral acid, organic acid, or acid salt, in combination with a wetting agent and detergent, is employed to remove oxide, shop soil, oil, grease, and other contaminants from metal surfaces, with or without the application of heat. The distinction between acid cleaning and acid pickling is a matter of degree, and there is often some overlapping in the usage of these terms. In general, however, acid pickling refers to a more severe treatment for the removal of scale from semi-finished mill products, forgings, or castings; whereas, acid cleaning is the term most frequently used when the acid solution is employed for final or near-final preparation of metal surfaces prior to plating, painting, or storage.

A variety of mineral and organic acids and solutions of acid salts are in common use, either with or without surfactants (wetting agents), inhibitors, and solvents. The very large number of formulations that have been used may be classified with respect to composition into five classes:

1. Inorganic (mineral) acid solutions
2. Organic acid solutions
3. Mixtures of (1) and (2)
4. Acid-solvent mixtures
5. Solutions of acid salts

Many acid cleaners are available as proprietary compounds, either as a liquid concentrate or a powder ready to mix with water.

Organic acids such as citric, tartaric, acetic, oxalic, and gluconic, and acid salts such as ammonium persulfate, sodium acid sulfate, and bifluoride salts, are used in various combinations. Solvents, such as ethylene glycol, monobutyl ether, and alkyl aryl polyether alcohol, and wetting agents, detergents, antifoam agents, and inhibitors may be included to enhance the removal of soil, oil, and grease.

Strength of the acid solutions varies from as weak as pH 5.5 for acid salt mixtures to the equivalent of the strong acids used for pickling.

Chromic acid solutions used occasionally to clean cast iron and stainless steel are composed of 8 ounces CrO_3 , 8 ounces H_2SO_4 , and 8 ounces HF per gallon of water for use at room temperature in an immersion system.

Another solution, used frequently for cleaning stainless steel, contains nitric acid (10 to 50 percent by volume) and hydro-fluoric acid (1 to 3 percent by volume). The steel is immersed in the solution at room temperature for 3 to 30 minutes.

Chromic acid solutions and mixtures containing chromic acid are often used as final rinses in acid cleaning - phosphating systems. The acid enhances the corrosion resistance of the coated surface and enables the rinse to clean excess and unattached phosphate from the surface of the treated metal without leaving hard water salts deposited on the surface.

Chromic acid is used in solutions of low pH when a strong oxidant is required; nitric acid is also a strong oxidant and a 10 - 20 percent nitric acid solution is used to brighten stainless steel.

Citric acid is used to clean rust from iron and steel without attacking the metal. This acid is also used in concentrations of 5 percent or less in formulas specifying sulfuric, phosphoric, and other commonly used acids, to enhance cleaning action. Cleaning is usually done at elevated temperatures (140-200° F) at concentrations ranging from 1/2 to 2 pounds per gallon. To a great extent, the solutions used in alkaline cleaning depend on their deter- sive properties for cleaning action and effectiveness. Agitation of the solu- tion and movement of the work pieces through it, although important, are secondary in their effect.

The principal methods employed in alkaline cleaning are soak, spray, and electrolytic. Other methods are variations incorporating the essential features of these three.

Soak cleaning, unless the soil is easily removable, is slower and less efficient than spray and electrolytic methods. Generally, it is employed only for moderate or small volumes of work.

Spray cleaning combines the advantages of the deterative properties of the solution with the impinging action of sprays, which loosen the soil mechan- ically and therefore provide greater efficiency than the soak method for remov- ing tenacious soils. However, because of foaming problems, spray cleaners have lower deterative properties than soak cleaners.

Electrolytic cleaning gives the cleanest surfaces obtainable from con- ventional alkaline methods. The resulting surfaces are suitable for the most exacting plating requirements and for subsequent finishing operations on close-tolerance parts. The effectiveness of this method results from strong agitation of the bath by gas evolution and oxidation reduction reactions during electrolysis. Another important factor is that similar electrical charges are imparted to certain dirt particles and to the work, causing the dirt to be repelled from the work surface.

2.2.6 Detergent Cleaning

Soaps and synthetic detergents ("syndets") are added to cleaning com- pounds to lower surface and interfacial tensions, and so enhance cleaning action.

Soaps used in alkaline cleaners must be low in cost if the cleaners are to be competitively priced. For this reason, sodium resinate, a by-product of the Kraft papermaking process, is extensively used instead of the common animal-fat soaps such as sodium laurate, palmitate, or stearate.

Several grades of sodium resinate are commercially available. The higher and lower grades are usually avoided, and middle grades are used most. Higher grades are more costly, as a result of their refining, but produce only slightly better cleaning than the middle grades. The low grades contain a fairly high content of unsaponified fatty acids, which form insoluble calcium, magnesium, and iron soaps with hard water. They should be used with a water-softening agent.

In small amounts, resinate soaps do not lower surface and interfacial tensions as much as synthetic detergents, but the soaps cost much less. Resinates also emulsify certain soils more easily than do the syndets, thereby, justifying their use in alkaline cleaners. Most soap cleaners contain both resinates and syndets.

Synthetic detergents are extensively used as surface-active agents in soak cleaners because they are freer-rinsing than soaps, aid soil dispersion, and prevent resoiling. They are effective in softening hard water and in lowering surface and interfacial tensions. In addition, the syndets are less susceptible than soaps to contamination by acid residues.

2.2.7 Ultrasonic Cleaning

Ultrasonic energy can be used in conjunction with several types of cleaners, but it is most commonly applied to chlorinated hydrocarbon solvents, water, and water with surfactants. Ultrasonic cleaning, however, is more expensive than other methods because of higher initial cost of equipment and higher maintenance cost. Consequently, the use of this process is largely restricted to applications in which other methods have proved inadequate.

Typical areas of application in which ultrasonic methods have proved advantageous are as follows:

1. Removal of tightly adhering or embedded particles from solid surfaces
2. Removal of fine particles from powder metallurgy parts

3. Cleaning of small precision parts, such as those for cameras, watches, or microscope components
4. Cleaning of parts made of precious metals
5. Cleaning of parts with complex configurations, when extreme cleanliness is required
6. Cleaning of parts for hermetically sealed units

Despite the high cost of ultrasonic cleaning, it has proved economical for applications that would otherwise require hand operations.

Size of part is a limitation, although no definite limits have been established. The commercial use of ultrasonic cleaning has been limited principally to small parts, such as those indicated in the above examples. The process is used as a final cleaner only, after most of the soil is removed by another method.

2.3 FINAL CLEANING

Final cleaners are basically heavy duty alkaline cleaners, but are always employed with current. They are designed both for soil removal and metal activation. Usually final cleaners follow a precleaning step, but in many cases final cleaning alone will suffice.

The objective of final cleaning is to remove completely any residual soil and to activate the metal surface. Activation is usually obtained by using reverse current electrocleaning. The gas scrubbing action of the oxygen generated assists soil removal, while reverse current aids in its removal and prevents the deposition of any metallic film or non-adherent metallic particles.

Work containing heat treat, welding, or other oxides may require a double cleaning cycle. In such cases the work is usually precleaned, anodically electrocleaned, pickled to remove the oxide, final electrocleaned, and acid dipped to neutralize the alkaline film.

2.3.1 Anodic Electrocleaning

The work is made anodic (positive) in an alkaline electrocleaner using low voltage (3-12v) dc current. Current densities vary from about 10 to 150 amp/ft² depending on the metal being cleaned and the cleaning time.

Anodic electrocleaning is desirable for final cleaning wherever possible because the metal is actually dissolved as well as cleaned. This action removes metallic smuts and prevents the deposition of non-adherent metallic film. Hydrogen embrittlement is avoided by anodic cleaning.

Important controlling factors are current density, temperature, and concentration, particularly on nonferrous metals to avoid etching and tarnishing.

Reverse current alkaline cleaning is not recommended for aluminum, chromium, tin, lead, or other metals which are soluble in alkaline electrocleaners.

2.3.2 Cathodic Cleaning

The work is made cathodic (negative) and the same equipment, voltage, and current densities are generally used as described under anodic cleaning. Hydrogen is liberated at the surface of the work equal to twice the amount of oxygen at the anode. Therefore, twice the gas scrubbing is obtained as with anodic cleaning. For this reason, cathodic cleaning is sometimes employed as a precleaner followed by anodic cleaning.

The work is actually being plated in a direct current cleaner. Any positive material is attracted to, and may be reduced and deposited on the surface. Any film (metallic) deposited is usually non-adherent but difficult to detect and remove.

Work critical to hydrogen embrittlement should not be cleaned cathodically unless adequate steps are taken after processing to remove the hydrogen. Generally, heat treatment for one hour at 400° F, immediately after processing, will remove the embrittling effect of the hydrogen.

Direct current cleaning is used for the following applications:

1. To clean metals such as chromium, tin, lead, brass, magnesium, and aluminum, which are dissolved or etched by anodic cleaning.
2. To clean buffed nickel prior to chromium plate. Anodic cleaning would produce a passive film on the nickel, due to oxidation, which would prevent the deposition of bright chromium.

2.3.3 Periodic Reversal Cleaning

Periodic reverse (PR) electrolytic cleaning is used generally to remove smut, oxide, and scale from ferrous metals. Alkaline compounded materials containing sequestering or chelating agents are usually used. The work is made alternately cathodic and anodic, using dc current at 6 to 15v. Work may be cleaned on racks or in a barrel. Cleaning and scale removal are accomplished by incorporating the mechanism of alkaline cleaning and the use of reducing and oxidizing conditions, coupled with strong metal chelation.

One of the advantages of PR cleaning is to eliminate the acid on certain types of work (hinges) where entrapment of acid aggravates bleed-out after alkaline plating (brass, copper, zinc, cadmium, tin). Oxides may also be removed without the danger of etching or the development of smut usually encountered from acid pickling.

2.3.4 Phosphate-Free Cleaners

Presently, cleaners free of phosphates are enjoying widespread popularity. Phosphates are used in some cleaners and function as water softeners, rinsing aids, soil suspending agents, and detergency boosters. Unfortunately, however, they are the main cause for increased algae growth in our waters where they are discharged. This increased algae growth (eutropication) is of prime concern, since it is becoming an increasing nuisance to sportsmen, causes infested waters to have an obnoxious odor and unpleasant taste, and even can have a detrimental effect on fish because of its high biological oxygen demand. The major offenders are trisodium phosphate, sodium tripolyphosphate, tetrasodium and tetrapotassium pyrophosphates, and the so called "glassy" phosphates, such as sodium hexametaphosphate, for example.

2.3.5 Acid Dipping

Acid dipping is the process of immersing a metal in an acid solution to remove oxide, scale, and other acid soluble soil and also to activate the metal surface. Mineral acids are usually used; however, mixtures of acid salts are utilized to increase both scale removal and activation.

Acid dipping may be classified into two categories: pickling and activation. Pickling is used to remove scale and oxide, while activation acid dipping is used to neutralize the alkaline film and to dissolve any light oxide coating developed in the final cleaning.

All acid solutions should be selected to meet the following qualifications: (1) The metal surface should not be disturbed any more than desired and (2) the metal salt formed from the reaction between the metal and the acid must be water soluble. To illustrate the latter point, sulfuric acid would not be suitable for preparing lead or leaded brass for electroplating, because of the formation of insoluble lead sulfate which would form a film between the basis metal and the electrodeposited metal, causing poor adhesion.

DC current may be used to aid activation, increase scale removal, remove projecting metal fragments, or reduce (level) surface roughness. The work is made cathodic for activation and scale removal applications. The evolution of hydrogen at the surface of the metal provides gas scrubbing, which increases the rate of scale removal. The reducing action reduces or eliminates passivity, for example, on stainless steel or buffed nickel which is to be electroplated. Reverse current is used to remove protruding metallic slivers and to smooth rough surfaces. Highly concentrated acids are generally used for this application.

Inhibitors may be used in pickling, but their use in activation acid dipping is usually not recommended because of the possibility of absorbed films which may interfere with later operations, such as plating, resulting in poor adhesion or cloudy plate. The function of the inhibitor in pickling is to reduce the amount of attack in areas where the scale has been removed and to minimize hydrogen embrittlement.

2.4 CHEMICAL MILLING

This process is the reverse of electrodeposition processes, in that it removes unwanted metal by selective or nonselective chemical etching. Materials reported as successfully etched are steel, aluminum, copper, brass, bronze, beryllium, and titanium; but the process is believed to be probably applicable to any material except silver, gold, platinum, and lead.

In its present proprietary form the process is one of controlled-depth etching employing masks of organic coatings and tapes resistant to hot alkaline solutions, modified etching solutions which act uniformly without adverse effect upon the metal, and electronic measuring and controlling equipment.

Prior to masking, parts must be cleaned, depending on prior condition, by a thoroughgoing cycle including solvent degreasing, hot-alkaline cleaner, cold-water rinse, chromic-acid dip, and hot-water rinse.

Some parts may need no masking before etching, as in the case of formed parts, extrusions, or forgings to be reduced in weight by overall (nonselective) etchings.

Most parts, however, require masks to be placed over those surface areas from which metal is not to be removed. By careful mask design and application, etching can in a single operation produce complex contours, cuts of varying width, and comparatively sharp corners. Cuts of different depths can be produced on large areas by progressive removal of different sections of masking tape at appropriate intervals. Sections can be tapered.

2.5 ANODIZING

Anodizing of aluminum is used for its protective value as well as for ornamentation. The coating thickness usually varies between 0.0001 and 0.0005 inch and is frequently expressed in ampere-minutes. This is based on the number of minutes of anodizing time per 12 amp per square foot. Some of the automotive industry applications require as little as 120 amp-min coatings for satisfactory dyeing. Black is probably the most difficult color to produce consistently, and usually requires a longer anodizing period for best results. Black finishes are usually anodized for 40 minutes at 12 amp per square foot (480 amp-min) followed by a 30-minute dyeing period. Anodizing-solution temperature is a very critical factor in color matching. As the solution temperature increases, the anodize film becomes softer and more porous, enabling it to absorb the dye more readily, resulting in darker shades of color.

The anodizing bath as made up contains 15 to 16 percent by weight sulphuric acid maintained at 70 to 85°F in a lead-lined tank. Most alloys in a new anodizing bath will require a current density of 12 to 12.5 amp per square foot. The high-silicone alloys will require 21 to 24 volts to obtain this current density. The magnesium-aluminum casting series should be anodized at 9 amp per square foot. As the electrolyte is used, the aluminum content will increase, and the resistance also increases as a result. Hence, higher voltages will be required as the bath gets older. It has been common practice to operate the sulphate bath until 24 volts are required to obtain this current density. The magnesium-aluminum casting series should be anodized at 9 amp per square foot.

SECTION III. PROCESS CONTROL

Testing and control methods should be simple, rapid, and not require much apparatus. In practice, both the number and size of parts to be cleaned may vary within wide limits. The degree of contamination may change similarity, and the degree of cleanliness required will differ according to a number of factors.

Under the influence of changing conditions it is seldom possible to predict with any accuracy the length of time a cleaning solution can be used before it decreases in efficiency to the point where it is better discarded. Testing methods are, therefore, of importance.

3.1 TESTING OF BASE METAL

The type of metal, its uniformity in chemical composition, its structure and its freedom from pores and inclusions, etc., can be investigated by known methods. The next stage is the determination of the quality of the surface finish resulting from mechanical treatments such as grinding, polishing, sand blasting, etc., to which the parts have been subjected.

3.1.1 Surface Roughness

The roughness of the surface can be determined by microscopy, interference microscopy, or the stylus type profilometer. Roughness values of the order of 2 rms may be determined under the microscope with the aid of special objectives. The interference microscope makes it possible to determine fine roughness values between 0.02 and 1 rms. The stylus type instrument can measure surfaces between 0.1 and 125 rms.

3.1.2 Crack Detection

Cracks and similar surface faults can be detected by both chemical and acoustical testing methods. In the case of ferromagnetic materials, it is possible to apply an oil film which contains very fine iron particles to the surface and subject the part to a magnetic field. The iron particles adhere to cracks and other surface defects and render them visible.

3.1.3 Grease Detection

An important factor is the freedom of the surface from grease. The simplest testing method for this property depends on the fact that a surface which is covered by grease is not wetted by pure water while a grease-free metal surface is wetted uniformly without a water break.

3.2 ANALYSIS OF SOLUTIONS

Methods of analysis are frequently recommended by the suppliers of the individual conversion coating processes and chemicals and should be followed carefully.

The ingredients of the solutions must be present in the correct proportions to insure that the solution is in hydrolytic equilibrium. The determination of these equilibrium concentrations frequently requires refined methods of analysis. The measurement of pH either electrometrically, with the aid of pH paper or indicators, or by titration against standard sodium hydroxide solution in the presence of a suitable indicator is a relatively simple task.

Normal analytical methods are employed for the determination of other constituents of the solution, such as iron, zinc, manganese, and accelerators. Impurities in the solution that have a harmful influence on the properties of the coating can also normally be determined by conventional analytical techniques.

3.3 TESTING OF COATINGS

The essential characteristics of a finish which are of primary importance are:

1. Appearance
2. Thickness
3. Porosity
4. Corrosion-resistance
5. Adhesion
6. Other properties, e.g., stress, wear and abrasion resistance, hardness, composition and structure, flexibility.

3.3.1 Appearance

The appearance of a finish when visually examined is informative. For example, continuity of the coating, freedom from pits and local defects, and absence of blistering are important to ensure that the finish has an adequate life and will not deteriorate rapidly in use.

3.3.2 Thickness

To a very considerable extent the protective values of many coatings, and particularly of electrodeposits, are a function of their thickness and all specifications include a thickness requirement. The measurement of the thickness of an electrodeposit or coating can be carried out in several ways. The most accurate method is to prepare a section by cutting and polishing, after which the thickness is measured under a microscope. Other test methods, both destructive and non-destructive, are available.

3.3.3 Porosity

The porosity of a coating is less significant than its thickness from the durability point of view; since, in general, the degree of porosity decreases with increasing thickness.

Tests for porosity are based on the application of a reagent which will react with the basis metal through pores in the coating and yet will not attack the deposit itself. For determining the porosity of cathodic coatings, such as nickel, chromium, tin, silver, or copper on steel the "ferroxyl tests" has been extensively used. The ferroxyl solution (10 gm/1 potassium ferricyanide and 5 gm/1 sodium chloride) is applied to the coating by means of holding a dampened cloth to the part for approximately 10 minutes. Any pores present will show themselves as blue spots on the cloth.

3.3.4 Corrosion Resistance

Corrosion resistance and porosity are closely inter-connected, but some indication of the expected life of a protective coating is obviously desirable and a great deal of attention has been given to the development of accelerated tests which will give some indication of durability. Exposure tests of plated coatings have been carried out on an extensive scale, the specimens being suspended usually on an inclined frame in the type of atmosphere (e.g., urban, marine, industrial, etc.) in which it is desired to carry out the tests.

3.3.5 Adhesion

The adhesion of a coating is an important measure of its serviceability, but satisfactory tests are difficult to devise. One of the earliest and still the most common method of testing adhesion in electrodeposits is the burnishing test. It is carried out by rubbing a smooth round steel bar to and fro across the plated surface. Poor adhesion shows itself in the development of blisters at the area of rubbing.

Other tests for adhesion include: impact tests, supersonic tests, hardness, and wear resistance.

3.4 SAFETY

As with most chemical processes, certain safety and health hazards are associated with metal processing operations. Such physiological harm that may result to the worker arises when insufficient care is taken and may affect either specific organs or the whole organism.

Halogenated hydrocarbons used in vapor degreasers possess narcotic properties. If high concentrations are present in the air, fainting, muscular relaxation, or partial or total unconsciousness may result. Therefore, maximum concentrations present in the air should be kept below limiting safety values. The limiting safety values for specific compounds can usually be obtained from the manufacturer.

Acid and alkaline solutions, even in dilute forms, can cause serious injuries to the eyes and other portions of the human body. Therefore, operators should be protected with face shields, rubber boots, and aprons.

It is customary to provide eye fountains and showers adjacent to acid cleaning operations for use in case of accidents. Nonslip floor coverings in the vicinity of tanks or spray operations are recommended.

Precautions must be taken against cyanides entering the cleaning system to avoid the formation of deadly gas. If work that has been processed in cyanide is to be cleaned, it should first be rinsed in hot water, then cleaned only in a well ventilated system.

Electrolytic cleaning systems are potentially dangerous, and rubber shoes and gloves are especially recommended to protect operators working around these installations.

Mist from spray systems can be a health hazard. Mist formation increases with the amount of work in process, temperature, acidity of the solution, and current density in electrolytic cleaning. This mist contains all the ingredients of the solution. Adequate ventilation is recommended.

3.5 ENVIRONMENTAL CONTROL

The current drive by local, state, and federal governments on land, water, and air pollution places a direct impact on metal finishing processes. At the present time, specific guidelines for the treatment and disposal of used chemicals do not exist. It is the policy, however, of Marshall Space Flight Center to be conscious of the need to consider any unfavorable impact of operations on the environment and to initiate any preventive measures necessary. Guidelines to carry out federal, state, and local requirements are given in the Environmental Quality Manual, MM 8800.1, dated April 28, 1971. The manual is published by Organization Development Center Plans and Resources Office, Marshall Space Flight Center, Huntsville, Alabama.

Other applicable references are listed below:

1. Federal Laws, Regulations, etc.

EO 11507 - Prevention, Control, and Abatement of Air and Water Pollution at Federal Facilities

EO 11514 - Protection and Enhancement of Environmental Quality

PL 89-272 - Solid Waste Disposal Act

PL 90-148 - Clean Air Act

PL 33USC 466 - Clean Water Act

PL 91-190 - National Environmental Policy Act

Title 10, Part 20 - Code of Federal Regulations - Standards for Protection Against Radiation

Title 42 - Public Health, PHS-DHEW - Prevention, Control and Abatement of Air Pollution from Federal Government activities: Performance Standards and Techniques of Measurements.

FWPCA - Guidelines for Prevention, Control and Abatement of Water Pollution

NAPCA - Interim Guide of Good Practice for Incineration at Federal Facilities

CEQ - Interim Guidelines for Environmental Statements

OMB - Bulletin 71-3 - Proposed Federal Actions Affecting the Environment, August 31, 1970

EO 11472, May 29, 1969

2. NASA Policy and Regulations

NMI 8800.3A - Prevention, Control and Abatement of Water Pollution

NMI 8800.4 - Prevention, Control and Abatement of Air Pollution

NPD 8800.6 - Policy on Environmental Quality and Control

NMI 8800.7 - Guidelines for the Preparation of Environmental Statements required by the NEPA of 1969.

NASA Procurement Regulation Directive No. 69-15 - Safety and Health

NASA Safety Manual, NHB 1700.1, Par. 1416

3. State and Local Laws and Ordinances

Solid Waste Disposal Act of State of Alabama

4. U. S. Army and MICOM Regulations and Instructions

U. S. Army Regulation (AR) 11-21, dated 3 November 1967, as amended

U. S. Army Missile Command MIR Number 420-5, as amended

SECTION IV. ALUMINUM ALLOYS

This section contains the process data related to aluminum alloys generated and used by S&E-PE-M Division of the S&E-PE Laboratory. Copies of specifications, standards, drawings, and publications required by contractors with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer. The chemical processes in this section are listed below.

<u>Paragraph</u>	<u>Title</u>
4.1	<u>SOLVENT CLEANING</u>
	Vapor Degreasing of Metallic Surfaces - MS 150.0
	The Cleaning of Spheres for the C-1 Space Vehicle - M-ME-MPROC 150.8
	Sulfuric Acid Strip of Space Vehicle Parts - MPD 25008
4.2	<u>CLEANING AND DEOXIDIZATION OF ALUMINUM ALLOYS</u>
	Deoxidation of Aluminum Alloys - MS 100.2A
	Sand and Vapor Blasting - MS 150.1
	Surface Treatment - M-ME-MPROC 150.7B
	Cleaning of Aluminum Alloy Weld Joints - MSFC PROC-463
	Cleaning and Deoxidation - MPD 25016A
	Deoxidation Prior to Welding - MPD 25020A
	Cleaning Prior to Welding - MPD 26607
	Cleaning ATM Components - MPD 26618
4.3	<u>CLEANING TO CRYOGENIC SPECIFICATIONS</u>
	Cleaning Liquid Hydrogen Cryogenic Tank for Space Shuttle - MPD 25023

Cryogenic Foam Insulation Application - MPD 38004

Cleaning of Cryogenic Insulation Test Container and Components -
M-ME-MPROC 101.6

4.4 CLEANING FOR USE IN ADHESIVELY BONDED STRUCTURES

Cleaning Prior to Application of Spray Foam - MPD 25017

Process Control for Application of Urethane Foam - MPD 38001

4.5 CONVERSION COATING AND ANODIZING

Anodizing Aluminum Alloys - MS 150.2A

Application of Conversion Coating - MS 150.3

4.6 CHEMICAL MILLING OF ALUMINUM ALLOYS

Specifications for Chemical Milling of Aluminum Alloys - MS 550.0

Chemical Milling of Bulkhead Gores - M-ME-MPROC 551.0

4.7 ELECTROLYIC CLEANING AND PLATING

Electro Cleaning of Space Vehicle Parts - MPD 26613

Cadmium Plating - MS 120.0

Nickel Plating - MS 120.1

Chromium Plating - MS 120.2

Electroless Nickel Plating - MS 120.3

Copper Plating - MS 120.4

Use of Marking Inks - MS 150.5

4.8 CLEANING OF TUBING AND FLEXIBLE HOSE

Cleaning of Flared and Unflared Flexible Tubing - M-ME-MPROC-
100.8E

Cleaning of Aluminum Tubing Ends - MPD 25010

4.9 APPLICATION OF DRY FILM LUBRICANTS

Specifications for Application of Dry Film Lubricants - MS 170.5

Application of MLF-5 Dry Film Lubricant - MPD 26621

4.10 VACUUM DRYING

Drying in a Vacuum - MS 101.0

4.1 SOLVENT CLEANING

MS 150.0

MANUFACTURING SPECIFICATIONS FOR VAPOR DEGREASING OF METALLIC SURFACES

1. SCOPE

1.1 Scope - This manufacturing specification covers the approved PE Laboratory requirements for vapor degreasing of metallic surfaces for use in space launch vehicles.

1.2 Applicability - The process set forth in this manufacturing specification is applicable to the following metals and alloys:

- Aluminum and Aluminum Alloys
- Stainless Steels, Carbon Steels, and Alloy Steels
- Magnesium and Magnesium Alloys
- Titanium and Titanium Alloys

1.3 Limitations - The following items shall not be vapor degreased:

- primed or painted components
- cables
- bearings or components containing bushings
- non-metallic components with the exception of those made of Teflon or Kel-F
- any component from which the organic insulating material has not been removed

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents of the issue in effect on the date of this specification form a part of this specification to the extent indicated herein.

SPECIFICATIONS

Federal

O-T-634a	Trichloroethylene, Technical
BB-N-411a	Nitrogen
UU-T-81D	Tags, Shipping, and Stock
PPP-T-60(2)	Tape; Pressure Sensitive Adhesive, Waterproof

Military

MIL-A-00148B(ORD) Aluminum Foil

George C. Marshall Space Flight Center

10509305	Cleanliness of Components for Use in Liquid Oxygen, Fuel, and Pneumatic Systems, Specifications for
MS101.0	Manufacturing Specification for Drying of Components in a Vacuum

(Copies of specifications, standards, drawings, and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

3. REQUIREMENTS

3.1 General - No deviation from the requirements of this manufacturing specification shall be permitted without prior written approval of the MSFC initiating activity.

3.2 Materials

3.2.1 Vapor-cleaning solvent - The trichloroethylene used for vapor-degreasing under the provisions of this specification shall meet all the requirements of Type II of Specification O-T-634a.

3.2.2 Liquid-cleaning solvent - The liquid cleaning solvent used for spot applications, immersion, or spray applications shall meet the requirements for Type I or II of Specification O-T-634a.

3.2.3 Gas, drying or preservation - Gases used in drying or preservation processes shall be filtered to a 100 micron level (absolute), the oil content shall not be greater than 3 parts per million by weight, and the moisture content shall not be greater than 24 parts per million by volume.

3.2.4 Aluminum foil - Dry annealed, aluminum foil used to protect cleaned surfaces shall conform to paragraph 6.3.2 or Specification MIL-A-00148B (ORD).

3.2.5 Tape - Tape used to secure and reinforce protective coverings shall conform to Specification PPP-T-60 (2).

3.2.6 Tags - Components cleaned by this process shall be identified by tags conforming to Specification UU-T-81D (1), Type C.

3.3 Procedure

3.3.1 Preliminary cleaning - Components to be vapor-degreased shall be inspected for foreign matter, stains, and undue concentrations of either difficult to remove or readily soluble material that would handicap uniform cleaning by vapor degreasing. These concentrations are to be removed by spot, immersion, or spray application of liquid trichloroethylene, or other suitable solvents, necessary to accomplish their removal.

3.3.2 Racking - Components to be cleaned shall be racked or supported in such a manner as to insure:

- That complete drainage of the condensate occurs.
- That no damage occurs to components by snagging on steam coils or other tank obstructions.

3.3.3 Immersion - Lower the racked components into trichloroethylene vapors 188° F ($\pm 3^\circ$) at approximately 11 feet per minute. Allow the vapors to condense and flow from the component until the component reaches the temperature of the vapor. At this point condensation will cease.

3.3.4 Removal - Remove from the vapors at a rate not greater than 11 feet per minute.

3.3.5 Foreign Matter - If visible solid particles adhere to the component after removal from the tank, spray the component inside the tank with liquid trichloroethylene pumped from the condensate collector.

3.3.6 Unclean parts - If the operator finds upon visual examination that the components are not thoroughly cleaned, repeat the operations in paragraphs 3.3.1 to 3.3.5 until parts are clean.

3.3.7 Drying - The method of drying shall be in accordance with the manufacturing specification covering the item to be cleaned. The components shall be checked visually to insure that no vapor film remains on the surface.

3.3.7.1 Air drying - Degreased components may be statically air-dried, when degreasing is merely a preparatory operation to subsequent alkaline cleaning or to a subsequent manufacturing operation.

3.3.7.2 Gaseous drying - Degreased, completed space launch vehicle components, which require no additional manufacturing or cleaning operations, shall be dried with nitrogen or air preparatory to preservation and packaging. Gases used for such drying shall meet the requirements of paragraph 3.2.3 of this specification. Drying shall be accomplished at 180°F ($\pm 20^\circ$) for 30 to 60 minutes at a gas pressure of 60 to 80 pounds per square inch measured at the item being dried.

3.3.7.3 Vacuum drying - Degreased components, such as flexible hoses, may be dried in a vacuum oven in accordance with MS 101.0.

4. QUALITY ASSURANCE PROVISIONS

4.1 Tank Solvent Protection - When the tank is not to be used for a prolonged period, the tank shall be covered.

4.2 Contaminants - Water, acids, metal chips, dirt, and other foreign materials that contribute to the decomposition of the solvent should be avoided.

4.3 Tank Solvent Control - When the work load is heavy, analyze the solvent at least once a week to determine the equivalent HCL content. If the work load is light, periodically determine the pH and temperature of the hot, Type II trichloroethylene.

4.3.1 pH control - When the pH (Hydrogen-Ion Concentration) falls below 7.0, chemically analyze the sump for equivalent HCL content. If the equivalent HCL content is 0.02 percent by weight or greater, the trichloroethylene shall be discarded.

4.3.2 Temperature limit - If the temperature of the tank sump reaches or exceeds 195 °F, discard the trichloroethylene.

4.4 LOX-Service Analyses - If vapor degreasing of a part or component is the last step prior to LOX, fuel, or pneumatic service installation, the effluent from a trichloroethylene rinse must be analyzed for particle and non-volatile residue content in the manner specified in paragraph 4.4.2 of MSFC Specification 10509305 to assure compliance with the requirements of paragraphs 3.6.1.1 and 3.6.1.2 of MSFC Specification 10509305. Components that fail the inspection tests of paragraph 4.4.2 of MSFC Specification 10509305 shall be rejected and reprocessed in accordance with paragraphs 3.3.2 to 3.3.4.1, inclusive, of this specification and retested as required above. See paragraph 6.2 for safety precautions to be observed in the handling and use of trichloroethylene.

5. PREPARATION FOR DELIVERY

5.1 Finished Parts - Degreased and dried, completed LOX components shall be packaged and preserved as required by MSFC Specification 10509305. When secured closures are specified in MSFC Specification 10509305 in lieu of aluminum foil to prevent contamination, the materials used shall be compatible with the cleaned LOX component.

5.2 Marking - When applicable, the tags attached to cleaned finished components shall record the following information:

- Part of identification number.
- Contractor identification.
- Method of cleaning and particle size (micron level).
- Date of cleaning.
- Service medium or intended use of component.
- Manufacturer's serial number.
- Any other information required by the contract.

6. NOTES

6.1 Intended Use - This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use in the vapor degreasing of metallic surfaces for use in space launch vehicle applications.

6.2 Safety Precautions - Trichloroethylene rinsing of parts or components for the purpose of obtaining particle or nonvolatile residue samples shall be done under a fume or exhaust hood in order to minimize breathing of trichloroethylene vapors. Rubber gloves shall be worn to prevent skin contact with liquid trichloroethylene. If no suitable fume or exhaust hood is available, personnel taking trichloroethylene samples shall wear respirators of a type approved by the Safety Office.

NOTICE: When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodians

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING PROCESS FOR THE CLEANING OF SPHERES FOR THE C-1 SPACE VEHICLE

1. SCOPE

1.1 Scope

This manufacturing process covers the approved Process Engineering Division requirements for cleaning and re-cleaning of all spheres presently being used in Saturn C-1 vehicles.

1.2 Applicability

The cleaning of spheres shall be accomplished by the processes outlined in this manufacturing process. Each cleaning and re-cleaning process covers a particular sphere. Note: Re-cleaning procedures are shown as separate processes.

1.3 Spheres

1.3.1 Type I - Lined

1.3.1.1 Aluminum alloy - epoxy resin-lined

A. Drawing No. 20M00414

- Clean by Process No. 1, paragraph 3.4.1 to cleanliness levels of MSFC-SPEC-164.

- Re-clean by Process No. 2, paragraph 3.4.2 to cleanliness levels of MSFC-SPEC-164.

1.3.1.2 Fiberglass - rubber-lined

A. Drawings (single units) 10438152, 10438153, and 10438154.

- Clean by Process No. 3, paragraph 3.4.3 to cleanliness levels of Drawing No. 10419907.

● Re-clean by Process No. 5, paragraph 3.4.5 to cleanliness levels of Drawing No. 10419907.

B. Drawing (Triplex Assembly) 10438020

● Clean by Process No. 4, paragraph 3.4.4 to cleanliness levels of Drawing No. 10419907.

● Re-clean by Process No. 6, paragraph 3.4.6 to cleanliness levels of Drawing No. 10419907.

1.3.2 Type II

1.3.2.1 Titanium

A. Drawings No. 20M00445 and 20M00449

● Clean and re-clean component by Process No. 7, paragraph 3.4.7 to cleanliness levels of Drawing No. 10419906.

2. APPLICABLE DOCUMENTS

2.1 Governmental

The following documents of the issue in effect on the date of use of this process form a part of this process.

SPECIFICATIONS

Federal

0-A-51	Acetone, Technical
0-E-760	Ethyl Alcohol (Ethanol), Denatured Alcohol and Proprietary Solvent
0-T-634	Trichloroethylene, Technical
BB-N-411	Nitrogen
BB-C-101	Carbon Dioxide

PPP-T-60	Tape; Pressure Sensitive Adhesive, Water-proof for Packaging and Sealing
UU-T-81	Tags, Shipping and Stock
O-P-94	Paper, Test, pH Indicator

Military

MIL-A-00148	Foil, Aluminum
MIL-D-3716	Desiccant
MIL-D-16791	Detergents, Non-ionic

George C. Marshall Space Flight Center

10419906	Cleanliness Levels, Cleaning and Inspection Procedure for Component Parts of Gas Bearing and Slosh Measuring Systems, Specification for
10419907	Spheres, Fiberglass, High Pressure
MSFC-SPEC-237	Solvent - Freon Precision Cleaning Agent, Specification for
MSFC-SPEC-164	Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic Systems, Specification for

DRAWINGS

George C. Marshall Space Flight Center

10438152	High Pressure Sphere 1.0 cubic foot
10438153	High Pressure Sphere 1.5 cubic feet
10438154	High Pressure Sphere 0.5 cubic foot
10438020	Container, High Pressure Assembly 5310 cubic inches
10M01076	Sphere, Metallic, High Pressure
20M00649	Slosh Measuring

20M00414	Envelope Bottle, Storage
20M00445	Bottle, Gas Storage 1728 cubic inches
20M00449	Bottle, Gas Storage 50 cubic inches
20M00645	Gas Bearing

3. REQUIREMENTS

3.1 General

No deviation from the requirements of this manufacturing process shall be permitted without prior written approval of the Process Engineering Division.

3.2 Materials

3.2.1 Vapor Cleaning Solvent

Trichloroethylene used for vapor-degreasing shall meet the requirements of Type II of Specification 0-T-634.

3.2.2 Liquid Cleaning Solvent

The liquid trichloroethylene used for immersion or flushing shall meet the requirements of Type I of Specification 0-T-634, with the further requirement that the non-volatile residue shall not be greater than 0.010 grams per 500 ml. There shall be no particle greater than 175 microns in any dimension and no more than 5 particles from 100 to 175 microns in size per 500 ml of solvent.

3.2.3 Drying or Preservation Gases

Nitrogen BB-N-411, or air, used for the drying or preservation of components shall contain no particle larger than 10 microns in any dimension. The oil content shall not be greater than 0.3 parts per million by weight.

3.2.4 Demineralized Water

Demineralized water shall contain no particles greater than 100 microns. It shall have a minimum electrical resistivity of 50,000 ohms. The pH of the water shall be between 6.0 and 8.0.

3.2.5 Aluminum Foil

Dry annealed foil used to protect surfaces shall conform to paragraph 6.3.2 of Specification MIL-A-00148 (ORD).

3.2.6 Tape

Tape used to secure protective coverings shall conform to Specification PPP-T-60.

3.2.7 Non-ionic Detergent

The detergent used in these processes shall be non-ionic and meet the requirements of Specification MIL-D-16791.

3.2.8 Tags

Tags shall be in conformance with UU-T-81. See paragraph 3.6.

3.2.9 Alcohol

The alcohol used in cleaning shall conform to Specification 0-A-396 and be pre-filtered to a 100 micron level.

3.2.10 Freon

Precision Cleaning Agent, MSFC-SPEC-237. This material shall be pre-filtered to a 10 micron, or lower, cleanliness level.

3.2.11 Acetone

Prefiltered acetone, used in cleaning, shall conform to Specification 0-A-51 to a 100 micron level.

3.3 Equipment

3.3.1 Particle Checking Equipment

3.3.1.1 Millipore filter holder assembly - Cat. No. XX 1004700 or equivalent.

3.3.1.2 Liter vacuum flash.

3.3.1.3 Forceps with unserrated tips.

3.3.1.4 Plastic disposable petri dishes.

3.3.1.5 Plastic filter paper holder.

3.3.1.6 Aspirator or other suitable source of vacuum.

3.3.1.7 Microscope with mechanical stage, measuring eye piece (ocular micrometer), and capable of magnification of 45X and 90X.

3.3.1.8 Microscope lamp - 5,000 candle power minimum.

3.3.1.9 Whatman 42 filter paper or approved equivalent.

3.3.2 Cleaning Equipment

3.3.2.1 Tank for hot detergent solution.

3.3.2.2 Pump to deliver hot detergent solution at 20-30 psig.

3.3.2.3 Spray lances with nozzles designed to fit the following spheres:

- Drawing No. 20M00414.
- Drawing No. 10438152, 10438153, and 10438154.
- Drawing No. 10438020.
- Drawing No. 20M00445 and 20M00449.

3.3.2.4 Whitfield bench, or approved equivalent.

3.3.2.5 Stands for spheres listed in paragraph 3.3.2.3.

3.3.3 Non-Volatile Residue Test Equipment,

3.3.3.1 Hot plate.

3.3.3.2 Weighing bottles.

3.3.3.3 Analytical balance (0.00005g)

3.3.3.4 Dessicator.

3.3.3.5 Oven with automatic temp. control and vent to atmosphere.

3.3.3.6 800 ml beakers.

3.3.3.7 Wash bottles (glass).

3.4 Cleaning Processes

3.4.1 Process No. 1, Cleaning, 20M00414 storage bottle.

3.4.1.1 Particle Size Limitation

Epoxy-resin lined spheres used in fuel or pneumatic systems shall be cleaned to comply with the cleanliness levels of MSFC-SPEC-164.

3.4.1.2 Cleaning Process

A procedure to meet the levels specified herein for epoxy-resin lined spheres is as follows:

A. Flush interior of spheres thoroughly for 20 to 30 minutes with a 5 parts detergent to 1000 parts water solution at a temperature of 122°F ($\pm 5^\circ$) by adding solution at the bottom of the component and allowing it to overflow at the top.

B. Flush interior of spheres from 5 to 18 minutes with demineralized water at room temperature.

C. Sample rinse water for particle count. If sample meets particles limitations, proceed to next step. If particle count is unsatisfactory, repeat B.

D. Purge with warmed drying gas ($130^{\circ}\text{F} \pm 10^{\circ}$).

E. Sealing - Follow procedure outlined in paragraph 3.4.3.4 of this specification.

3.4.2 Process No. 2, Re-cleaning to 20M00414 Sphere Drawing.

3.4.2.1 Particle Size Limitation - Same as for Process No. 1, paragraph 3.4.1.1

3.4.2.2. Re-cleaning Process

A re-cleaning process to meet the levels specified herein for epoxy-resin lined metal sphere is as follows:

A. Spray with a solution of 5 parts detergent to 1000 parts demineralized water solution at a temperature of 120 to 140°F . Use a spray nozzle with lance size compatible with sphere opening at a water pressure of 20 to 30 psig for 15 to 20 minutes.

B. Spray rinse with demineralized water at ambient temperature to 140°F with a water pressure of 20 to 40 psig for 5 minutes, after all sudsing has ceased.

C. Sample rinse water for particle count. See paragraph 4.3.1 for method of taking sample.

D. Dry by purging with warmed drying gas ($130^{\circ}\text{F} \pm 10^{\circ}$) using a spray lance.

E. Sealing - Follow procedure outlined in paragraph 3.4.3.4 of this specification.

3.4.3 Process No. 3, Cleaning High Pressure Spheres 10438152, 10438153, 10438154.

3.4.3.1 Particle Size Limitation

Rubber-lined fiberglass spheres used in pneumatic systems of space vehicles shall have the interior of the spheres cleaned to comply with the cleanliness levels of Drawing No. 10419907.

3.4.3.2 Cleaning Procedure

A cleaning procedure to meet the cleanliness levels specified herein for rubber-lined, fiberglass spheres is as follows:

A. Washing - The spheres shall be filled to two-thirds their capacity with a 5 parts detergent to 1000 parts demineralized water solution at 122° F ($\pm 5^\circ$). The spheres shall be tilted so that the solution shall be agitated. Agitation shall be continued while the spheres are rotated one-half turn, at 15 minute intervals, for one complete revolution.

B. Draining and Flushing - The solution shall be drained from the spheres. While in the drain position the spheres shall be flushed out with a stream of warm (120 to 140° F), demineralized water. Flushing shall be continued for 5 minutes after all signs of sudsing have ceased. The spheres shall be allowed to drain for 5 minutes.

3.4.3.3 Drying

A. Water Removal - Approximately 2 liters of re-distilled ethyl alcohol, conforming to grade II, Class B of Specification 0-E-760, shall be poured into the spheres. The spheres shall be rotated in such a manner that the entire interior surface will be washed by the alcohol. The spheres shall be thoroughly drained. Approximately 2 liters of re-distilled acetone, conforming to Specification 0-A-51, shall be poured into the spheres. The spheres shall be rotated in such a manner that the entire interior surface will be washed with the acetone. The spheres shall be thoroughly drained.

WARNING: An explosive mixture now exists inside the sphere. Keep away from excessive heat or open flame, and guard against electrostatic discharge.

B. Pneumatic Purging - While the spheres are in the drain position, a stream of warm ($130^\circ\text{F} \pm 10^\circ$), clean, dry, oil-free gas (dew point minus 65 degrees F, or lower, with absolute particle level of 10 microns) shall be injected into the orifice. Media used shall be nitrogen (conforming to type I, Class I, grade B of Specification BB-N-411), or carbon dioxide (conforming to grade B, type I of Specification BB-C-101), or air. Pneumatic purging shall be conducted four times, using a 5-minutes-on, 5-minutes-off procedure for three consecutive cycles. The fourth 5-minute purge shall be conducted following a 24-hour waiting period.

3.4.3.4 Sealing

The spheres shall be allowed to cool for 2 hours. At the end of this period the orifice shall be sealed with a plug incorporating a holder containing desiccant material conforming to Specification MIL-D-3716. The plug shall permit adequate exposure of the desiccant to the interior air, but shall prevent contamination of the interior by desiccant particles larger than 100 microns.

3.4.4 Process No. 4, Clean (Refer to 10438020).

3.4.4.1 Particle Size Limitation

Rubber-lined, fiberglass, triplex sphere assemblies used in pneumatic systems of space vehicles shall have the interior of the spheres cleaned to comply with the cleanliness levels of Drawing No. 10419907.

3.4.4.2 Cleaning Procedure

A cleaning procedure to meet the cleanliness levels specified herein for rubber-lined, fiberglass, triplex sphere assemblies is as follows:

A. Washing - The sphere assembly shall be mounted in a vertical position (see Figure 1 of 10419907). The assembly shall be filled with a 5 parts detergent to 1000 parts demineralized water solution at 122° F ($\pm 5^\circ$). The solution shall be pumped through the assembly, from bottom to top, for 30 minutes.

B. Draining and Flushing - The solution shall be drained from the assembly. The assembly shall be flushed out with warm (120 to 140°F) demineralized water. Flushing shall be continued for 5 minutes after all signs of sudsing have ceased. The assembly shall be allowed to drain for 5 minutes.

3.4.4.3 Drying

A. Water Removal - Re-distilled ethyl alcohol, (see 3.4.3.3, paragraph A) shall be pumped through the assembly. Sufficient alcohol shall be used so that the entire interior surface will be washed by the fluid. The assembly shall be thoroughly drained. Re-distilled acetone (see 3.4.3.3, paragraph A) shall be pumped through the assembly. Sufficient acetone shall be used so that the entire interior surface will be washed by the fluid. The assembly shall be thoroughly drained.

WARNING: An explosive mixture now exists inside the assembly. Keep assembly away from excessive heat or open flame, and guard against electrostatic discharge.

B. Pneumatic Purging - Pneumatic purging shall be as specified in 3.4.3.3, paragraph B.

3.4.4.4 Sealing

Sealing of the assembly shall be as specified in paragraph 3.4.3.4.

3.4.5 Process No. 5, Recleaning Spheres 10438152, 10438153, and 10438154.

3.4.5.1 Particle Size Limitation

Rubber-lined fiberglass spheres used in pneumatic systems of space vehicles shall have the interior of the spheres cleaned to comply with the cleanliness levels of Drawing No. 10419907.

3.4.5.2 Re-Cleaning Procedure - A re-cleaning procedure to meet the cleanliness levels specified herein for rubber-lined, fiberglass spheres, is as follows:

A. Washing - Spray with a solution of 5 parts detergent to 1000 parts demineralized water solution at 120 to 140° F. Use a spray nozzle with lance compatible with sphere opening at a water pressure of 20 to 30 psig for 15 to 20 minutes.

B. Draining and Flushing - Spray rinse with demineralized water at ambient temperature to 140° F with a water pressure of 20 to 40 psig for 5 minutes, after all sudsing has ceased.

C. Sample rinse water for particle count. See paragraph 4.3.1 for method of taking sample.

3.4.6.3 Drying

A. Pneumatic Purging - Dry by purging with warmed drying gas (120 to 140° F) to meet requirements of paragraph 3.2.3.

3.4.6.4 Sealing

Sealing of the assembly shall be as specified in paragraph 3.4.3.4.

3.4.7 Process No. 7, Cleaning and Re-Cleaning of Titanium Gas Storage Bottles 20M00445 and 20M00449.

3.4.7.1 Particle Size Limitation

Titanium spheres used in Gas Bearing or Slosh Measuring Systems shall have the interior of the sphere cleaned to comply with the cleanliness levels of Drawing No. 10419906.

3.4.7.2 Non-Volatile Residue

The requirements for this test are the same as stated in Drawing No. 10419906.

3.4.7.3 Cleaning Procedure

The procedure outlined in the following paragraphs will provide an acceptable cleanliness level prior to final cleaning. There shall be no testing requirement during this stage of cleaning, provided the steps listed below are followed in order. A cleaning procedure to meet the cleanliness levels specified herein for titanium spheres will be accomplished as follows:

A. Washing - Spray with a solution of 5 parts detergent to 1000 parts demineralized water at 120 to 140° F. Use spray nozzle with lance size compatible with sphere opening at a water pressure of 20 to 60 psig for a period of 15 minutes.

B. Rinse - Spray rinse with demineralized water at 120 to 140° F using a spray nozzle with a water pressure of 40 to 60 psig. Continue rinsing action for 5 minutes after all signs of sudsing have ceased.

C. Drying - Dry with warm (120 to 140° F) gas conforming to paragraph 3.2.3 using a spray lance.

3.4.7.4 Final Cleaning

The final cleaning procedure necessary to produce a cleaned sphere to a 20 micron absolute particle level as required in paragraph 3.4.7.1 is accomplished in the following steps:

A. Spray rinse the sphere, which is mounted in a Whitfield bench on a tripod arrangement, using a minimum of 5 volumes of Freon Precision Cleaning Agent so that the critical surface area is thoroughly washed with the fluid.

B. Sample the test fluid for particle contamination and non-volatile residue as in paragraphs 4.3.1 and 4.3.2

C. Dry the component with warm (120 to 140° F) gas conforming to paragraph 3.2.3, using a spray lance.

3.4.7.5 Sealing

Seal sphere openings with two layers of precleaned aluminum foil and secure with tape.

3.5 PACKAGING

Components that have been cleaned and dried acceptably and are not scheduled for immediate use should be preserved and unit packaged immediately after drying to reduce the possibilities of recontamination. All openings shall be protected by a covering of a minimum of two layers of aluminum foil, whether parts are scheduled for immediate use or for storage. The covering shall be secured with tape which conforms to Specification PPP-T-60. Do not wrap the sphere in polyethylene film.

3.6 IDENTIFICATION

Unless otherwise specified, cleaned components shall be identified with tags that conform to Specification UU-T-81-type C, which shall provide the following information:

- Part and identification numbers
- Contractor identification

- Method of cleaning and particle size, micron level
- Date of cleaning
- Service medium or intended use of component

4. ACCEPTABILITY PROVISIONS

4.1 GENERAL

The supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the Government. Inspection records of the examination and tests shall be kept complete and available to the Government as specified in the contract or order. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.2 ACCEPTANCE TEST

Unless otherwise specified by the procuring activity, the acceptance test shall be performed on all spheres submitted for acceptance. The acceptance test shall be performed in such a manner so that the sphere will not be recontaminated.

4.2.1 Visual Examination

All critical surfaces of cleaned components shall be visually examined for the presence of corrosion, grease, dirt, oil, or any other foreign matter. The presence of visible contamination shall be cause for rejection.

4.3 TESTS

4.3.1 Particle Size Analysis

All critical surfaces shall be tested in a manner to trap the test solvent after assuring that the solvent has rinsed all surface areas. After completion of the rinsing process, a sample of the solvent shall be filtered through Whatman Number 42 filter paper, or approved substitute, into a clean, degreased beaker. A minimum of 500 ml of solvent shall be collected as a sample.

4.3.1.1 Particle Size

Examine the residue trapped on the filter paper with a microscope to determine conformance to the specific cleanliness requirements for the various cleaning and re-cleaning processes.

4.3.2 Non-Volatile Residue (Except non-metallic components)

The used test solvent contained in the beaker shall be examined for non-volatile residue as follows:

A. Carefully clean all glassware to be used by washing in hot water containing detergent, rinsing 3 times with demineralized water, 3 times with pre-filtered ethanol, and 3 times with pre-filtered 'Freon' Precision Cleaning Agent, all meeting the requirements of this specification.

B. Dry an aluminum foil dish of weight less than 10 mg in an oven from 95 to 105° C until constant weight is reached, and weigh to the nearest 0.01 mg.

C. Fill a clean 800 ml beaker three-fourths full from a liter sample of the cleaning agent.

D. Place the beaker on a hot plate, cover with a watch glass suspended on a glass rod support, and gently boil cleaning agent until approximately 20 ml of the sample remain in the beaker.

E. Continue adding portions of the 1-liter sample to the beaker and boil off the solution until all the sample has been evaporated.

F. Wash the residue from the beaker into the tared aluminum dish with the cleaning agent from a Guth-type wash-bottle equipped with a 1.20 micron Millipore filter. Rinse several times for complete removal of residue.

G. Evaporate the washings in the aluminum dish in a hot air bath or on a hot plate adjusted to low temperature.

H. After complete evaporation, dry the dish in an oven at a temperature within a range from 95 to 105° C for one-half hour (minimum).

I. Remove the dish from oven, cool and reweigh to the nearest 0.01 mg.

J. Calculate residue by the following formula:

$$\frac{(A - B) \times 10^3}{W} = \text{ppm residue}$$

Where: A = weight of dish and residue (in mg)

B = weight of empty dish

W = weight of sample (in grams)

4.4 SOLVENT CONTROL CHECKS

4.4.1 Particle Size

Whenever applicable, a control test shall be conducted on cleaning liquids to determine conformance to the applicable specification or drawing.

4.5 DRYING

Immediately following cleanliness testing, as applicable, the sphere shall be dried as specified in the cleaning process being followed.

4.6 RECLEANING

Components that fail the visual inspection of 4.2.1 or the tests of particle size and non-volatile residue, as applicable, shall be recleaned by the applicable process.

5. NOTES

5.1 SURFACE AREA

The standard unit of area used for determination of contamination levels in 1.0 square foot of critical surface area (area that will normally contact the service medium). Exact calculations of surface area are not mandatory.

5.2 This manufacturing process has been coordinated within the Process Engineering Laboratory and is intended for use in the cleaning, testing, and protection of spheres of all types when specified as components of a C-1 space vehicle.

5.3 Any deviation from this basic procedure will require written concurrence from the Methods Research and Development Branch, M-ME-M. An emergency concurrence may be made by telephone, but it must be confirmed in writing.

Preparing Activity
Process Engineering Laboratory
George C. Marshall Space Flight Center

PROCESS CONTROL AND OPERATING PROCEDURE FOR
SULFURIC ACID STRIP OF SPACE VEHICLE PARTS

1. SCOPE

1.1 This manufacturing process data covers the necessary precautions and procedures for process control of sulfuric acid strip of space vehicle parts.

2. EQUIPMENT AND MATERIALS

2.1 Equipment:

- Handling fixtures
- Pipette
- Erlenmeyer flask
- Watch glass
- Steam bath
- Burettes

2.2 Materials:

- Sulfuric acid strip bath sample
- Phenolphthalein indicator
- Standard sodium hydroxide
- Potassium dichromate
- Concentrated sulfuric acid
- Distilled water
- Ferro ammonium sulfate

3. PROCEDURES

3.1 Solution control and operating conditions.

3.1.1 Determine sulfuric acid content:

- (a) Pipette 5.0 ml sample into a 250 ml erlenmeyer flask.
- (b) Add 40.0 ml distilled water and a few drops of 2 percent phenolphthalein in alcohol solution.
- (c) Titrate with standard 1.0 N sodium hydroxide.
- (d) Calculations:

Total sulfuric acid percent =

$$\frac{N \text{ of NaOH} \times (49) \times 0.134 \times 100}{5 \text{ ml}}$$

3.1.2 Glycerine Determination

- (a) Pipette 10 ml of solution into a 500 ml erlenmeyer flask containing 0.7456 grams of dry potassium dichromate, 10 ml of water, and 15 ml concentrated sulfuric acid.
- (b) Cover the flask with a watch glass and heat in a steam bath for 2 hours at a temperature of 93 to 100° C.
- (c) Cool and titrate with standardized ferrous ammonium sulfate (300 g/liter with 100 ml of sulfuric per liter) using potassium ferricyanide as an outside indicator.
- (d) Run a blank test the same with omission of the sample.
- (e) Calculations:

$$\frac{0.2 (\text{blank titration} - \text{sample titration})}{\text{Blank titration}} = \% \text{ glycerine}$$

4. CONTROL

4.1 Check normality of solution against known standard once per week or as often as solutions are made-up.

4.2 Insure that beakers, pipettes, watch glass, and all laboratory instruments are maintained in a clean condition.

MANUFACTURING SPECIFICATION FOR
THE DEOXIDATION OF ALUMINUM ALLOYS

1. SCOPE

1.1 Scope - This manufacturing specification covers the approved requirements for cleaning and deoxidation of aluminum alloys for resistance welding and other applications, excluding aluminum honeycomb, in space launch vehicles.

1.2 Applicability - The cleaning and deoxidation procedures set forth in this specification are applicable to types 5086, 5456, 6061, 7075, 2014, and 2219 aluminum alloys having surfaces which are not painted, anodized, alodined, or coated with any kind of protective material other than a naturally occurring oxide film.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents of the issue in effect on the date of this specification form a part of this specification to the extent indicated herein.

SPECIFICATIONS

Federal

PPP-T-60	Tape, Pressure Sensitive Adhesive, Waterproof for Packaging and Sealing
QQ-A-283a	Aluminum Alloy, Plate and Sheet, 7075
QQ-A-327b	Aluminum Alloy, Plate and Sheet, 6061
O-T-634a	Trichloroethylene, Technical

Military

MIL-A-00148B (ORD)	Aluminum Foil
JAN-A-489	Acetone (For Ordnance Use)
MIL-A-19070A (Ships)	Aluminum Alloy, Plates and Sheets, 5086 (K186)
MIL-A-19842 (Ships (1))	Aluminum Alloy, Plates and Sheets, 5456

STANDARDS

Federal

Federal Test Method Standard 151a	Metals; Test Methods
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PUBLICATIONS

George C. Marshall Space Flight Center

M-F&AE-M-1-60	Evaluation of Turco 4215 Alkaline Cleaner
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3. REQUIREMENTS

3.1 General - No deviation from the requirements of this specification shall be permitted without the prior written approval of the MSFC Initiating Activity.

3.2 Alloy Composition - The aluminum alloys to which this specification applies shall be of the compositions specified in the documents referenced in paragraph 2.1.

3.3 Cleaning and Deoxidizer Solutions

3.3.1 Trichloroethylene - The trichloroethylene used in vapor degreasing aluminum alloys shall be Type II of Specification O-T-634a.

3.3.2 Alkaline Cleaner (Turco 4215, or equal) - The alkaline cleaner bath shall be maintained at a concentration of 9 to 15 ounces of cleaner per gallon of solution, with 12 ounces per gallon of solution the optimum concentration, and at a pH of between 7.1 and 9.3 inclusive.

3.3.3 Deoxidizer (Turco Smut-Go No. 1, or equal) - The deoxidizer bath shall be maintained at a concentration of 12 to 16 ounces of deoxidizer per gallon of solution at a pH of 3 to 5.

3.4 Cleaning and Deoxidizing Procedure

3.4.1 Preclean - Using a clean cloth or steel wool saturated with acetone, remove all inked lettering, wax pencil or crayon markings, grease, heavy oils, and forming lubricants such as Lubri-Plate or wax. Scrub all surfaces of the aluminum.

3.4.2 Alkaline clean - Immerse the dried material in an alkaline, non-silicated cleaner (Turco 4215, or equal) containing 9 to 15 ounces of cleaner per gallon of solution at 185°F ($\pm 5^\circ$) for 25 to 35 minutes.

3.4.3 Spray rinse - Spray rinse with ambient temperature tap water while slowly raising and removing the part from the 4215 alkaline cleaner tank.

3.4.4 Dip rinse - Rinse in hot (170 - 180°F) tap water for a minimum of 10 minutes. If water breaks appear on material surfaces after rinsing, repeat steps 3.4.2 through 3.4.6 until water breaks no longer appear.

3.4.5 Deoxidize - Immerse the alkaline cleaned material in a mildly acidic deoxidizer (Turco Smut-Go No. 1, or equal) containing 12 to 16 ounces of deoxidizer per gallon of water at room temperature for the times given below:

6061

45 minutes

All others

20 to 30 minutes

3.4.6 Rinse - Rinse deoxidized material in deionized water (50 000 ohms specific resistance) at room temperature for a minimum of 10 minutes with constant agitation. Subsequently, rinse with hydro air gun using deionized water and missile grade air.

3.4.7 Dry - Allow material to statically dry.

3.4.8 Protect - Completely wrap and seal all fabricated parts or components in two thicknesses of dry annealed aluminum foil, sealed and reinforced with tape, immediately after drying to prevent contamination during necessary handling and transportation. Items too large to be packaged and sealed shall be handled only with clean rubber or cotton gloves after drying and prior to resistance welding or other fabrication operations.

3.5 Cleanliness of Aluminum Alloys - Immediately after deoxidation, cleanliness of aluminum alloys shall be such that the average of any surface resistance readings taken shall not exceed 15 microhms and no single resistance reading shall be greater than 20 microhms.

4. MANUFACTURING CONTROL PROVISIONS

4.1 Solution Concentrations

4.1.1 Alkaline cleaner (Turco 4215, or equal) - At least once each week, a quantitative analysis of the alkaline cleaner bath shall be run to insure that the concentration specified in paragraph 3.3.2 is being maintained. At least once each day the pH of the alkaline cleaner bath shall be determined to insure that it is within the limits specified in paragraph 3.3.2. Proprietary materials and methods recommended by the manufacturer shall be used in the checking of proprietary products.

4.1.2 Deoxidizer (Turco Smut-Go No. 1, or equal) - At least once each week a quantitative analysis of the deoxidizer bath shall be run to insure that the concentration specified in paragraph 3.3.3 is being maintained. At least once each day the pH of the deoxidizer bath shall be determined to insure that it is within the limits specified in paragraph 3.3.3. Proprietary

materials and methods recommended by the deoxidizer manufacturer shall be used in the checking of proprietary products.

4.2 Determination of Material Cleanliness

4.2.1 At least four 4-inch by 24-inch test panels (any two of which shall be considered as a test set) of the same gage aluminum alloy as the material being cleaned and deoxidized shall be processed with, and in exactly the same manner as, each different alloy of each thickness of aluminum processed during a work day.

4.2.2 Within one hour after drying, one test set, which represents a basket of deoxidized aluminum alloy parts or a skin segment weldment, shall have its surface resistance measured by means of a Double Kelvin Bridge Surface Resistance Meter. The remaining test set, consisting of two panels, shall be used as a check for comparison.

4.2.2.1 A surface resistance reading shall be made for each 2 square inches of test set panel surface. (Total of 48 readings for a 4-inch by 24-inch test panel set.)

4.2.2.2 The average of all surface resistance readings taken on a test panel set shall not be greater than 15 micro-ohms and no single resistance reading shall be greater than 20 micro-ohms.

4.2.3 In the event that a test panel set does not meet the surface resistance requirements specified in paragraph 4.2.2.2, the entire lot of material represented by that test panel set shall be reprocessed through the cleaning and deoxidation steps outlined in paragraph 3.4, together with the same four 4-inch by 24-inch test panels; after which the surface resistance of the two test panels sets shall again be checked as specified in paragraph 4.2.2.1. This reprocessing of material and test panels and rechecking of test panel sets shall continue until the test panel sets meet the surface resistance requirements of paragraph 4.2.2.2.

4.3 Resistance Welding - After cleaning and deoxidation, material shall be resistance welded within 48 hours. Surface resistance shall not exceed 50 micro-ohms at the time of resistance welding.

5. PREPARATION FOR DELIVERY

5.1 Packaging - Each deoxidized part or component shall be wrapped

in dry annealed aluminum foil, or other suitable oil and paraffin free protective material (Saran wrap, etc.), to prevent recontamination during handling and storage prior to installation.

6. NOTES

6.1 Intended Use - This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use in the chemical deoxidation of aluminum alloys, excluding aluminum honeycomb, preparatory to their use in space launch vehicle applications.

NOTICE - When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government Procurement operation, the United States Government thereby incurs no responsibility nor obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation.

MANUFACTURING SPECIFICATIONS FOR
SAND AND VAPOR BLASTING

1. SCOPE

1.1 Scope - This manufacturing specification covers the approved Process Engineering Laboratory requirements for sand and vapor blasting space vehicle component surfaces.

1.2 Applicability - The requirements set forth in this specification are applicable to sand or vapor blasting of components fabricated from the following materials:

- Stainless steel alloys - 304, 316, 321, 410, 17-7PH, and 19-9DL
- Aluminum alloys - 2014, 2219, 5456, 6061, and 7075
- Ferrous metal parts
- Teflon
- Glass fiber laminates

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this specification, form a part of this specification.

SPECIFICATIONS

Federal

PPP-T-60	Tape, Pressure Sensitive Adhesive, Waterproof - For Packaging and Sealing
O-T-634A	Trichloroethylene, Technical

Military

MIL-P-17667A	Paper, Wrapping, Chemically Neutral
MIL-S-13165A	Shot Peening of Ferrous Metal Parts
MIL-S-17726A	Sand, Sandblast
MIL-S-22262	Sand, Sandblast, and Grain, Abrasive; Ship Hull Blast Cleaning

George C. Marshall Space Flight Center

MS150.0	Manufacturing Specification for Vapor Degreasing of Metallic Surfaces
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2.2 Other Publications - The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

Turco Products, Incorporated

Bulletin No. 78 Turco 4215

(Applications for copies should be addressed to Turco Products, Inc., Southeast Regional Offices, 200 Ottley Drive N.E., P. O. Box 13585, Station K, Atlanta 24, Georgia.)

3. REQUIREMENTS

3.1 General - No deviation from the requirements of this specification shall be permitted without prior written approval of the MSFC Initiating Activity.

3.2 Dimensions - All parts shall be within dimensional tolerance before blasting. (See paragraph 3.5.1.)

3.3 Equipment

3.3.1 Vapor degreasing equipment

3.3.2 Alkaline cleaning tank

3.3.3 Vapor blasting equipment - Pangborn Hydro-Finish or equivalent.

3.3.4 Sand blasting equipment - Vacu-Blast or equivalent.

NOTE: Size of blasting equipment necessary shall be determined by the size of the parts to be blasted.

3.3.5 Ventilation equipment - Ventilation equipment shall be provided for the blasting room and for the blasting equipment.

3.3.6 Oil and moisture trap - An oil and moisture trap shall be provided in the compressed air line supplying sand or vapor blasting equipment.

3.4 Materials

3.4.1 Solvent degreaser - Trichloroethylene Type II or approved equal.

3.4.2 Alkaline cleaner - Turco 4215 or approved equal.

3.4.3 Blasting grit

3.4.3.1 Garnet of 60, 80, 100, or 250 mesh as applicable.

NOTE: Separate stocks of grit must be maintained for non-corrosion resistant steel and for corrosion resistant metals such as stainless steel and aluminum.

3.4.4 Compressed air - Compressed air must pass through an oil and water trap during blasting.

3.4.5 Tape - Type 3, Class 1.

3.4.6 Rubber or plastic plugs - Rubber or plastic plugs may be used to mask holes in part being blasted.

3.5 Preparation of Parts for Blasting

3.5.1 Fabrication - Perform fabricating operations prior to blasting whenever practical. Heat treatment operations shall be performed before blasting and final machining shall be performed after blasting to the maximum extent possible.

3.5.2 Degrease and clean - Remove all oil and grease completely from metallic components by vapor degreasing (Trichloroethylene type II or approved equal). Part may be cleaned with an alkaline cleaner after vapor degreasing if necessary. (Reference MS150.0.)

3.5.3 Protection - Protect surfaces which are not to be blasted with a Type 3, Class 1 tape. Holes in part to be blasted shall be covered with rubber or plastic plugs.

3.6 Personnel

3.6.1 Qualification - Each operator shall be fully instructed in the operation of the equipment and in the provisions of this specification before blasting any parts.

3.7 Precautions

3.7.1 Separate abrasives - Do not use the same grit on non-corrosion resistant steel that is used on aluminum and stainless steel. (See NOTE in paragraph 3.4.3.1.)

3.7.2 Contaminated abrasives - Use only abrasive that is free from contaminants for sand and vapor blasting. Only dry abrasive shall be used for sand blasting. Abrasive visibly contaminated with grease or oil shall be discarded.

3.8 Detailed Blasting Procedure

3.8.1 General

3.8.1.1 Nozzle shall be held as nearly perpendicular as possible to surface of the part being blasted.

3.8.1.2 Blast systematically for complete coverage. Move nozzle in a straight line when possible, overlapping by 50 percent or more on each pass.

3.8.1.3 Nozzle must be turned away from part until equipment is operating in the proper manner.

3.8.1.4 Nozzle must not be directed continuously at the same area on any part for more than 5 seconds, as overheating and erosion of the part would occur in that area causing deleterious effects.

NOTE: Safety precautions are outlined in paragraph 3.10.

3.8.2 Steel Alloys

3.8.2.1 For sand blasting, use garnet abrasive of 60 to 100 mesh. For vapor blasting, use aluminum oxide or garnet abrasive of 100 to 250 mesh.

3.8.2.2 Use selectively regulated air pressure to prevent damage to the component to be cleaned.

3.8.2.3 Adjust feed of grit to approximately 7 lb/min.

3.8.2.4 Maintain approximately 6 to 18 inch distance between nozzle and work.

3.8.2.5 Maintain linear rate of travel of nozzle across work at approximately 80 ft/min whenever practical.

3.8.3 Aluminum Alloys

3.8.3.1 For sand blasting, use garnet abrasive of 60 to 100 mesh. For vapor blasting, use aluminum oxide or garnet abrasive of 100 to 250 mesh.

3.8.3.2 Use approximate air pressure of 50 psi.

3.8.3.3 Adjust feed of grit to approximately 7 lb/min.

3.8.3.4 Maintain approximately 6 to 18 inch distance between nozzle and work.

3.8.3.5 Maintain linear rate of travel of nozzle across work at approximately 50 ft/min whenever practical.

3.8.4 Teflon

3.8.4.1 For sand blasting, use aluminum oxide abrasive of 60 to 100 mesh. For vapor blasting, use aluminum oxide abrasive of approximately 200 mesh or garnet of approximately 250 mesh.

3.8.4.2 Use approximate air pressure of 50 psi.

3.8.4.3 Adjust feed of grit to approximately 7 lb/min.

3.8.4.4 Maintain approximately 30-inch distance between nozzle and work.

3.8.4.5 Maintain linear rate of travel of nozzle across work at approximately 50 ft/min.

3.8.5 Glass fiber laminates

3.8.5.1 Use 36 grit garnet blasting abrasive for sand blasting.

3.8.5.2 Use approximate air pressure of 50 psi.

3.8.5.3 Adjust feed of grit to approximately 7 lb/min.

3.8.5.4 Maintain approximately 30-inch distance between nozzle and work.

3.8.5.5 Maintain linear rate of travel of nozzle across work at approximately 50 ft/min.

3.9 Processing After Sand or Vapor Blasting

3.9.1 Cleaning - All parts after sand or vapor blasting and prior to subsequent treatment shall be free of corrosion, blasting debris, grease, oil, and other foreign matter. Remove blasting debris as soon as possible after blasting. This is particularly important with vapor blasting since discoloration of metal can take place immediately. A suction device or clean, dry compressed air will be used to remove sand blasting debris. Vapor blasting debris is removed by washing with hot tap water and drying with compressed air. Further cleaning may be accomplished according to the appropriate specification.

3.9.2 Handling - Blasted areas on parts shall be handled only with dry, clean cloth gloves or by means of suitable clean holding devices.

3.9.3 Protection - To avoid contamination, each part shall be separately wrapped in clean, dry, sulphur-free wrapping paper conforming to Specification MIL-P-17667A.

3.10 Safety Precautions

3.10.1 Vapors - Observe normal respiratory precautions when using volatile solvents.

3.10.2 Dust - Ventilating equipment must be operating at all times during sand blasting to combat the heavy dust formation.

3.10.3 Safety equipment - For sandblasting large articles or during prolonged exposure in the blasting room, the operators shall be provided with special clothing and respirators which completely protect them from the dusty atmosphere.

WARNING: Overexposure could result in silicosis or other respiratory or pulmonary disease. The safety officer shall make the final determinations for precautions.

4. MANUFACTURING CONTROL PROVISIONS

4.1 General - To insure uniform smoothness and cleanliness of the blasted part, continuous visual inspection shall be performed. Skilled workmanship and frequent in-process examinations can eliminate surface imperfections such as eroded areas and areas missed during blasting. After blasting, parts shall be subjected to 100 percent visual inspection. Approval of the process, materials, and equipment implies no guarantee of acceptance of the final sand or vapor blasted part; items shall conform to all applicable requirements of this specification.

5. PREPARATION FOR DELIVERY

5.1 Preservation and Packaging - When there is to be a delay in subsequent treatment of a part, a covering shall be provided for the part. Clean, dry, sulfur-free wrapping paper is sufficient. (See paragraph 3.9.3.)

6. NOTES

6.1 Intended Use - This manufacturing specification, developed by the Process Engineering Laboratory of the George C. Marshall Space Flight Center, is intended for use in sand and vapor blasting stainless steel alloys, aluminum alloys, ferrous alloys, Teflon, and glass fiber laminates intended for use in space launch vehicle applications.

NOTICE - When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use,

or sell any patented invention that may in any way be related thereto.

Custodian

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING PROCESS FOR
THE SURFACE TREATMENT OF SKIN AND BULKHEAD MATERIAL
AND FULLY ASSEMBLED CONTAINER FOR THE S-1C LAUNCH VEHICLE

1. SCOPE

1.1 Scope - This manufacturing process covers the approved Process Engineering Laboratory requirements for the cleaning and surface treatment of skin, bulkhead material, and fully assembled container of the S-1C launch vehicle fuel and LOX containers prior to welding.

1.2 Applicability - The cleaning and surface treatment procedures outlined in this manufacturing process are applicable to 2219 aluminum alloy of the following heat treatment conditions: T-31, T-37, T-81, T-87, T352, and T-852.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this process, form a part of this process.

SPECIFICATIONS

Federal

0-T-634	Trichloroethylene, technical
0-E-760	Ethyl Alcohol (Ethanol); Denatured Alcohol; and Proprietary Solvent

Military

MIL-C-5541	Chemical Films for Aluminum and Aluminum Alloys
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George C. Marshall Space Flight Center

MS 100.2	Deoxidation of Aluminum Alloys
MS 150.0	Vapor Degreasing of Metallic Surface
MS 150.1	Sand and Vapor Blasting
MS 150.3	Application of Conversion Coatings to Aluminum Alloys

STANDARDS

Federal

151 A	Metals, Test Methods
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2.2 Other Publications - The following documents form a part of this process. Unless otherwise indicated, the issue in effect on the date of issuance of this process shall apply.

Allied Research Products, Inc.

No. 14-2 Al Coat Bulletin	Operating Data for Iridite No. 14-2 Al Coat for Aluminum and Aluminum Alloys
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(Copies of this publication may be obtained from Allied Research Products, Inc., 4404 E. Monument St., Baltimore 5, Maryland)

Turco Products, Inc.

Bulletin No. 78	Turco 4215 (Non-Silicated Alkaline Spray Washing and Hot Tank Compound for use on Aluminum)
Bulletin No. 84	Turco Smut Go (2897 Redstone)

(Copies of these publications may be obtained from Turco Products, Inc., P. O. Box 1055, Wilmington, California)

3. REQUIREMENTS

3.1 Equipment

3.1.1 Vapor degreasing equipment - Aerojet General Corporation trichloro-ethylene vapor degreasing equipment with working clearance of 24 feet length, 6 feet width, and 10 feet vapor depth shall be used.

3.1.2 Sand blasting equipment - Vacu-Blast equipment capable of blasting with 200 mesh or finer glass beads at 35 to 55 psi air pressure shall be used.

3.1.3 Alkaline cleaning tanks - Tanks 24 feet long, 6 feet wide, and 10 1/2 feet deep, constructed of mild steel, may be used for cleaning parts. The welded construction shall include joints welded from inside and outside the tank.

3.1.4 Deoxidizer or conversion coating solution tanks - Tanks 24 feet long, 6 feet wide, and 10 1/2 feet deep, constructed of stainless steel or of hot-rolled, S.A.E. 1020, plate steel covered inside with Koroseal, rubber, or other acid resistant coating and with joints welded inside and outside may be used.

3.1.5 Final rinse tanks - The rinsing tank immediately following the conversion coating tank may be constructed of hot rolled, S.A.E. 1020, plate steel with joints welded inside and outside. The inside surfaces may be protected with Plastisol. Dimensions are 24 feet length, 6 feet width, 10 1/2 feet depth.

3.1.6 Crane - An overhead rail-type crane system of approximately 2000 pounds capacity may service the tank area.

3.1.7 Racks - Racks for supporting parts during cleaning and conversion coating shall permit thorough draining and may be constructed of stainless steel.

3.2 Materials

3.2.1 Vapor-degreasing solvent - Trichloroethylene conforming to Specification 0-T-634, Type II, shall be used for vapor or liquid degreasing.

3.2.2 Alkaline cleaner - The alkaline cleaner shall be Turco 4215, at a concentration of 6 to 12 ounces per gallon, at a temperature of 140 to 180°F, and at a pH of 8 to 10.

3.2.3 Deoxidizer - The deoxidizer employed shall be Turco Smut - Go (2897 Redstone) at a concentration of 8 to 16 ounces per gallon of water, at ambient temperature and a pH of 2 to 4.

3.2.4 Final Rinse - Demineralized filtered water, possessing a minimum electrical resistivity of 50,000 ohms, shall be used for all rinsing and conversion coating solutions.

3.2.5 Conversion coating solution - Iridite 14-2 A1 coat at a concentration of 0.75 to 1.75 ounces per gallon of aqueous solution with a pH between 1.2 and 1.9 at ambient temperatures shall be used. Adjust pH with nitric acid when conversion coating solution pH rises above 1.9.

3.2.6 Blasting abrasive - Contamination-free glass beads, 200 mesh or finer shall be used.

3.3 Procedures - Parts not requiring artificial aging shall be conversion coated following the forming and/or milling operation. Parts to be artificially aged after welding (2219-T37 gore segments) shall be conversion coated after artificial aging.

3.3.1 Cleaning - Surface shall be cleaned as follows:

3.3.1.1 Solvent cleaning - Vapor degrease with Type II trichloroethylene at 188°F ($\pm 3^\circ$) by lowering part into vapors at approximately 11 feet per minute. Allow the vapors to condense and flow from the skin or bulkhead material until the component reaches the temperature of the vapor. Remove the component at a rate not greater than 11 feet per minute.

3.3.1.2 Alkaline cleaning - Alkaline clean with Turco 4215 by dip immersion for 20 minutes at 140 to 180°F. The concentration of cleaner shall be 6 to 12 ounces per gallon of water and at a pH of 8 to 10.

3.3.1.3 Rinse - Rinse with hot (160 to 180°F) filtered tap water until pH of aluminum surface is between 6 and 8. If surface is not water-break free, repeat paragraphs 3.3.1.1 and 3.3.1.2.

3.3.1.4 Deoxidation - Deoxidize with Turco Smut-Go (2897 Redstone) for 30 minutes at a concentration of 8 to 16 ounces per gallon of water at ambient temperature at a pH of 2 to 4.

3.3.1.5 Rinse - Rinse with ambient temperature demineralized water until pH of aluminum surface is between 6 and 8.

3.3.2 Conversion coating - Conversion coat with Iridite 14-2 by dip immersion for 1 1/2 to 3 minutes at ambient temperature. The bath shall contain 0.75 to 1.75 ounces of Iridite 14-2 per gallon of water with a pH between 1.2 and 1.9.

3.3.2.1 Rinse - Rinse with ambient temperature demineralized water until pH of aluminum surface is between 6 and 8.

3.3.2.2 Drying - Allow to dry for a minimum of 24 hours at ambient temperature.

3.3.3 Conversion Coating of Fully Assembled Container.

3.3.3.1 Prior to spray conversion coating, the surface should be inspected for abraded areas. If such areas are found, they should be treated as follows:

A. Mechanically clean abraded areas using stainless steel wire brush or aluminum oxide abrasive paper.

B. Solvent wipe with alcohol or acetone.

3.3.3.2 Spray Conversion Coating.

A. Spray detergent solution (0.1 percent Dowell F-33 in demineralized water) at 130 to 140°F for several hours at 160 to 200 gpm and 200 to 300 psi.

B. Spray rinse with demineralized water at 130 to 140°F, 160 to 200 gpm, and 200 to 300 psi.

C. Spray with solution of Iridite 14-2 (1/2 to 2 oz/gal.) at ambient temperature, 160 to 200 gpm, and 200 to 300 psi for 2 to 4 minutes.

D. Spray rinse with ambient temperature demineralized water at 160 to 200 gpm and 200 to 300 psi.

E. Dry for several hours using filtered air heated to 130 to 140° F.

F. Purge with missile grade air at 130 to 140°F for 20 to 60 minutes or until the effluent air meets quality specifications.

3.3.4 Stripping of Parts Requiring Artificial Aging - Conversion coated parts which require subsequent artificial aging (such as 2219-T37) shall have the coating entirely removed in the following manner:

3.3.4.1 Immerse parts in Turco Form Etchant No. 13 at ambient temperature until the conversion coating is entirely removed. Metal removal shall not exceed 0.0006 inch.

3.3.4.2 Rinse parts thoroughly by immersion at ambient temperature water agitated by air.

3.3.4.3 Deoxidize with Turco Smut-Go (2897 Redstone) for 30 minutes at a concentration of 8 to 16 ounces per gallon of water at ambient temperature at a pH of 2 to 4.

3.3.5 Stripping of Weld Areas - Parts not requiring subsequent artificial aging (such as 2219-T87) shall have the conversion coating removed from the edge to 9 inches from the edge of a net trim part.

3.3.5.1 If there is a delay between the conversion coating operation and the stripping of weld areas, preclean the area to be stripped as follows:

A. Wipe with clean, dry, lint free cloth.

B. Solvent wipe with ethyl alcohol or acetone.

3.3.5.2 Remove the conversion coating from the weld areas by one of the following methods:

A. Use contamination-free glass beads, 200 mesh or finer, for blasting of conversion coating from the surface. Cleaning speed should be approximately 2 sq ft/min at 35-55 psi air pressure.

B. Use stainless steel wire brush or stainless steel wool to remove the conversion coating.

4. MANUFACTURING CONTROL PROVISIONS

4.1 Visual Examination - Coatings usually exhibit an iridescent color ranging from light golden to brown. The coating shall be continuous, smooth, adherent, and uniform in appearance.

4.2 Salt Spray Test - Conversion coatings applied to 2219 aluminum alloy test panels, coated simultaneously with skin and bulkhead material, shall withstand 168 hours exposure (MIL-C-5541) when tested per Federal Standard 151A.

4.3 pH Determination - All determinations shall be made electrometrically using a potentiometer with a glass electrode to maintain the pH ranges in paragraphs 3.2.2, 3.2.3, and 3.2.5.

4.4 Cleanliness - The cleanliness of cleaned parts prior to conversion coating shall be checked by employing the water-break test outlined in paragraph 4.4.1.

4.4.1 Water-break test procedure - A mist of distilled water shall be atomized onto the surface under test by employing any convenient small atomizing devices. If the water gathers into discrete droplets within 25 seconds, and if the surface shows a water-break within that time, then the surface shall be considered as having failed the test. If the water forms a continuous film but flashes out suddenly over a large area, then this characteristic shall presume the presence of free alkali, residual detergent, or uncleanness, and the surface shall be considered as also having failed the test. If the water droplets coalesce into a continuous film of water without a sudden flash out and form a lens, then the surface shall be considered as having satisfactorily passed the water-break test.

5. PREPARATION FOR DELIVERY

5.1 General - The protection given parts coated with conversion coatings during handling or shipment of finished parts must be ample and adequate to insure acceptable parts. Parts shall be treated as required by purchasing agreements, drawings, or related specifications.

6. NOTES

6.1 Intended Use - This manufacturing process, coordinated within the Process Engineering Laboratory of the George C. Marshall Space Flight Center, is intended for the surface treatment of skin and bulkhead material and fully assembled container for the S-1C launch vehicle.

6.2 Any deviation from the basic procedure will require written concurrence from Process Engineering Laboratory. An emergency concurrence may be made by telephone, but must be confirmed in writing.

6.3 Caution - String, wood, paint, or other organic material shall not be placed in conversion coating solutions because of the strongly oxidizing nature of these solutions.

6.4 Packaging of finished parts shall be accomplished by the instructions in applicable engineering drawings.

Finished parts, prior to packaging, must be in a dry condition. The parts shall be retained in a dry condition while being packaged.

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

PROCEDURE FOR
CLEANING OF ALUMINUM ALLOY WELD JOINTS
FOR HIGH QUALITY WELDS

1. SCOPE

1.1 Scope - This procedure specifies the method that shall be used in cleaning aluminum alloy weld joints prior to welding where high quality welds are required.

1.2 Applicability - This procedure establishes a standard process applicable to final cleaning of aluminum alloy weld joints immediately prior to welding where high quality welds are required.

2. APPLICABLE DOCUMENTS

There are no applicable documents to this procedure.

3. RESPONSIBILITIES

3.1 Design activities - Each design activity shall be responsible for implementing the provisions of this procedure.

3.2 Quality control - The quality control or inspection personnel of each performing activity shall be responsible for assuring that the requirements of this procedure are met.

4. PROCEDURES

4.1 Environmental Control.

4.1.1 Cleaning - All final cleaning shall be accomplished in an environment which will not degrade the quality of the weld. The controlled environment shall be maintained until the welding operation has been completed.

4.2 Equipment - Equipment to be used shall be clean and free of any foreign matter which could be detrimental to weld quality.

4.3 Personnel instruction - Personnel involved in the cleaning procedure shall be instructed to exercise extreme caution at all times to reduce the possibility of contamination.

4.4 Precleaning - Component parts to be welded shall have all grease, oil, etc., removed from the entire surface prior to preparing the joints for welding.

4.5 Detail requirements.

4.5.1 Mechanical scraping - All surfaces (root and face sides) adjacent to the abutting edges of the weld joint shall be mechanically scraped for a distance of 1/2 inch back from the abutting edge. The scraped surfaces shall have a bright metallic appearance with no visible streaks of the dull aluminum oxide of the original surface.

4.5.2 Draw filing - The abutting edges shall be draw filed, using long strokes parallel to the abutting edge. The file shall be used to remove all burrs at the corners. Files shall be cleaned as necessary to prevent galling.

4.5.3 Vacuuming - The cleaned surface shall be vacuumed after cleaning, by using a clean, long bristle brush attachment to remove loose particles of matter. The type of brush and length of bristle shall be such that the weld quality is not damaged.

NOTE

Following mechanical cleaning of any surfaces, nothing should touch the cleaned surface (e.g., handling with gloves, degreasing solvents, wiping with lint-free cloths, etc.) except the vacuum cleaning brush.

4.6 Time lapse - The time lapse permissible between cleaning and welding is determined by the storage environment. Each situation must be determined on its own merits. In all cases, the welding operation shall be performed within the time lapse necessary to produce the required weld quality.

5. REPORTS

There are no applicable reports to this procedure.

6. MODIFICATIONS OR CHANGES

6.1 Recommendations for modifications or changes to the requirements of procedures specified herein shall be submitted in writing to the Chief,

Technical Writing and Editing Section, Astronautics Laboratory
for coordination with all MSFC activities having an
interest in the document.

NOTICE - When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian:

NASA - George C. Marshall Space Flight Center

Preparing Activity:

George C. Marshall Space Flight Center

PROCEDURE FOR CLEANING AND DEOXIDATION OF ALUMINUM ALLOYS

1. SCOPE

1.1 Scope - This manufacturing Procedure covers the requirements for the chemical cleaning and deoxidation of aluminum alloys.

1.2 Applicability - The procedures set forth in this document are applicable to Types 2219, 2014, 6061, 7075, 5086, and 5456 aluminum alloys having surfaces which are not painted or coated with any protective material other than a naturally occurring oxide film or in contact with a dissimilar metal alloy.

2. EQUIPMENT AND MATERIAL

2.1 Equipment

- Baskets
- Hoist
- Cleaning Tanks
- Spray nozzle

2.2 Materials

- Steel wool
- Clean Wiping Cloths
- Trichloroethylene, Type II per Spec O-T-634A
- Deionized Water
- Turco 4215, Non-silicated
- Deoxidizer Turco Smut-Go No. 1 or equal

- Tap water
- Acetone
- Gloves, Cotton or Rubber
- Dry Annealed Aluminum Foil

3. CLEANING

3.1 Using a clean cloth or steel wool saturated with acetone, or other suitable solvents, remove all inked lettering, wax pencil or crayon markings, grease, heavy oils, or forming lubricants.

3.1.1 For parts that are excessively corroded, mechanically remove the corrosion products by hand sanding. Material that requires the removal of conversion coating may be processed through the Turco 13 B Alkaline Cleaner.

3.2 Degrease by immersing material in a vapor degreaser containing trichloroethylene at 180°F ($\pm 5^\circ$) until vapors cease to condense on the surface of the material.

3.3 Statically dry the thoroughly degreased material.

3.4 Immerse the material in an alkaline, non-silicated cleaner, Turco 4215 or equivalent, at a concentration of 9 to 15 ounces per gallon of solution, (pH 7.1 to 9.3) at 170°F ($\pm 5^\circ$) for 30 (± 15) minutes.

3.5 Spray rinse with ambient temperature water while slowly raising and removing the part from the 4215 alkaline cleaner tank.

3.6 Dip rinse in agitated hot water until no water breaks appear on the surface of the material. If water breaks appear on surface of material, repeat steps 3.2 and 3.6 until no water breaks appear.

3.7 Immerse the rinsed material in an acidic deoxidizer (Turco Smut-Go No. 1 or Equal) containing 12 to 16 ounces of deoxidizer per gallon of solution at ambient temperatures for 30 (± 15) minutes.

3.8 Rinse deoxidized material in deionized water at ambient temperature for 5 to 10 minutes utilizing agitated rinse water.

3.9 Allow material to statically dry.

3.10 Wrap all parts in dry annealed aluminum foil or clean wrapping paper. Items too large to be packaged must be handled with clean rubber or cotton gloves.

4. CONTROLS

4.1 Vapor Degreaser - The normal operating procedures of maintaining proper temperatures, keeping the covers closed when not in use, etc., will apply.

4.2 Alkaline Cleaner - The strength and temperatures will be maintained within the critical specified limits during the operations described. Material that is badly corroded or extremely contaminated may be reprocessed thru steps 3.4 to 3.9. If, after reprocessing a second time, the material is still unacceptable, contact the project engineer for instructions.

DEOXIDATION OF ALUMINUM ALLOYS PRIOR TO RESISTANCE WELDING

1. SCOPE

1.1 Scope - This manufacturing specification covers the approved requirements for the cleaning and deoxidation of aluminum alloys for resistance welding and other applications, excluding aluminum honeycomb, in space launch.

1.2 Applicability - The cleaning and deoxidation procedures set forth in this specification are applicable to types 5086, 5456, 6061, 7075, 2014, and 2219 aluminum alloys having surfaces which are not painted, anodized, alodined, or coated with any kind of protective material other than a naturally occurring oxide film. All alloys are to have a RMS micro inch finish of 0-40.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Cleaning Tanks
- Spray Nozzle
- Double Kelvin Bridge Surface Resistance Meter
- Tally Surf or equivalent

2.2 Materials

- Turco 4215 Alkaline Cleaner
- Methylethylketone (MEK)
- Steel Wool
- Clean Cloth
- Turco Smut-Go No. 1
- Deionized Water

3. PROCEDURE

3.1 General - No deviation from the requirements of this specification shall be permitted without the prior written approval of S&E-PE-M, MSFC Initiating Activity.

3.2 Alloy Composition - The aluminum alloys to which this specification applies shall be of the compositions specified in the documents referenced in paragraph 1.2.

3.3 Cleaning and Deoxidizer Solutions

3.3.1 Trichloroethylene - The trichloroethylene used in vapor degreasing aluminum alloys shall be Type II of Specification O-T-634a.

3.3.2 Alkaline Cleaner (Turco 4215, or equal) - The alkaline cleaner bath shall be maintained at a concentration of 9 to 15 ounces of cleaner per gallon of solution, with 12 ounces per gallon of solution being the optimum concentration, and at a pH between 7.1 and 9.5 inclusive.

3.3.3 Deoxidizer (Turco Smut-Go No. 1, or equal) - The deoxidizer bath shall be maintained at a concentration of 12 to 16 ounces of deoxidizer per gallon of solution at a pH of 0.6 to 1.4.

3.4 Cleaning and Deoxidizing Procedure

3.4.1 Machined Parts Preclean - Using a clean cloth saturated with methylethylketone, remove all inked lettering, wax pencil or crayon markings, grease, heavy oils, and forming lubricants, such as Lubri-Plate or wax.

3.4.1.1 If necessary, scrub all surfaces of the aluminum with Turco 4215 using clean cloths on a stick or soft bristle brush until the surface is relatively water-break free.

3.4.1.2 Then rinse with clean water by rinse or hose-down techniques before the detergent dries.

3.4.2 Unmachined Parts Preclean

1. Wipe with Methylethylketone (MEK).

2. Immerse in Turco 4215 (Tank in Honeycomb Area) at 170° F ($\pm 10^\circ$) for 10 minutes.

3. Rinse in hot deionized water at 160° F ($\pm 10^\circ$) (Honeycomb Area).

4. Dip in Honeycomb Etch at 160° F ($\pm 10^\circ$) for 5 minutes.

5. Rinse in cold deionized water for 5 minutes.

3.4.3 Alkaline Clean - Immerse the material in an alkaline, non-silicated cleaner (Turco 4215, or equal) containing 9 to 15 ounces of cleaner per gallon of solution at 165° F ($\pm 10^\circ$) for 25 to 35 minutes.

3.4.4 Spray Rinse - Spray rinse with ambient temperature tap water while slowly raising and removing the part from the 4215 alkaline cleaner tank.

3.4.5 Dip Rinse - Rinse in hot (170 - 180° F) tap water for a minimum of 10 minutes. If water breaks appear on material surfaces after rinsing, repeat steps 3.4.1 through 3.4.4 until water breaks no longer appear.

3.4.6 Deoxidize - Immerse the alkaline cleaned material in an acidic deoxidizer (Turco Smut-Go No. 1, or equal) containing 12 to 16 ounces of deoxidizer per gallon of water at room temperature for the times given below:

6061	45 minutes
All others	20 to 30 minutes

3.4.7 Rinse - Rinse deoxidized material in deionized water (50 000 ohms specific resistance) at room temperature at a pH 7 (± 1) for a minimum of 10 minutes with constant agitation. Subsequently, rinse with hydro air gun using deionized water and missile grade air.

3.4.8 Dry - Allow materials to statically dry.

3.4.9 Protect - Completely wrap and seal all fabricated parts or components in two thicknesses of dry annealed aluminum foil, sealed and reinforced with tape, immediately after drying to prevent contamination during necessary handling and transportation. Items too large to be packaged and sealed shall be handled only with clean rubber or cotton gloves after drying and prior to resistance welding or other fabrication operations.

3.5 Cleanliness of Aluminum Alloys - Immediately after deoxidation only, cleanliness of aluminum alloys shall be such that the average of any

surface resistance readings taken shall not exceed 25 microhms and no single resistance reading shall be greater than 40 microhms.

4. MANUFACTURING CONTROL PROVISIONS

4.1 Solution Concentrations

4.1.1 Alkaline Cleaner (Turco 4215, or equal) - At least once each week, a quantitative analysis of the alkaline cleaner bath shall be run to insure that the concentration specified in paragraph 3.3.2 is being maintained. At least once each day, the pH of the alkaline cleaner bath shall be determined to insure that it is within the limits specified in paragraph 3.3.2. Proprietary materials and methods recommended by the manufacturer shall be used in the checking of proprietary products.

4.1.2 Deoxidizer (Turco Smut-Go No. 1, or equal) - At least once each week, a quantitative analysis of the deoxidizer bath shall be run to insure that the concentration specified in paragraph 3.3.3 is being maintained. At least once each day the pH of the deoxidizer bath shall be determined to insure that it is within the limits specified in paragraph 3.3.3. Proprietary materials and methods recommended by the deoxidizer manufacturer shall be used in the checking of proprietary products.

4.2 Determination of Material Cleanliness

4.2.1 At least four 4-inch by 4-inch test panels, any two of which shall be considered as a test set of the same gage aluminum alloy as the material being cleaned and deoxidized shall be processed with, and in exactly the same manner as, each different alloy of each thickness of aluminum processed during a work day.

4.2.2 Within one hour after drying, one test set, which represents a basket of deoxidized aluminum alloy parts or a skin setment weldment, shall have its surface resistance measured by means of a Double Kelvin Bridge Surface Resistance Meter. The remaining test set, consisting of two panels, shall be used as a check for comparison.

4.2.2.1 A surface resistance reading shall be made for each 2 square inches of test set panel surface.

4.2.2.2 The average of all surface resistance readings taken on a test panel set shall not be greater than 25 microhms and no single resistance reading shall be greater than 40 microhms.

4.2.3 In the event that a test panel set does not meet the surface resistance requirements specified in paragraph 4.2.2.2, the entire lot of material represented by that test panel set shall be reprocessed through the cleaning and deoxidation steps outlined in paragraph 3.4, together with the same four 4-inch by 4-inch test panels, after which the surface resistance of the two test panels sets shall again be checked as specified in paragraph 4.2.2.1. This reprocessing of material and test panels and rechecking of test panel sets shall continue until such time as the test panel sets meet the surface resistance requirements of paragraph 4.2.2.2.

NOTE

Clad materials may only be recleaned once.

4.3 Resistance Welding

After cleaning and deoxidation, material shall be resistance welded within 72 hours.

CLEANING OF S-1C BULKHEAD GORE SEGMENTS PRIOR TO WELDING (MERIDIAN)

1. SCOPE

1.1 This manufacturing process data covers the preparation of the bulkhead gore segment surfaces prior to being welded into an S-IC fuel or liquid oxygen system tank bulkhead.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Gloves
- Scrapers
- Files
- Stainless steel wool (non-magnetic)

2.2 Materials

- Alcohol - O-E-760
- Freon, Precision Cleaning Agent - MSFC - SPEC-237
- Aluminum foil, dry annealed - MIL-A-00148B
- Tack cloth
- Lint-free cloth
- Tape

3. PROCEDURE

3.1 Cleaning - The bulkhead gore segments shall be cleaned as follows:

3.1.1 Remove gross contamination from each bulkhead gore segment surface with a tack cloth. Do not use the tack cloth on the edge to be welded.

3.1.2 Wipe the area of each bulkhead gore segment to be welded with a clean lint-free cloth saturated with alcohol. Dry with a dry, lint-free cloth.

NOTE

Clean only the surface of the gores to be welded, except for the last gore segment. The last segment shall have all four surfaces to be welded prepared because the area will not be accessible for scraping prior to making the last weld.

NOTE

Clean gloves shall be worn when performing the following operations. Handling of contaminated tools with the gloves is not permitted.

3.1.3 Wipe the weld area of each bulkhead gore segment with a clean lint-free cloth saturated with Freon precision cleaning agent. Dry the weld area by wiping with a dry, lint-free cloth.

3.1.4 Scrape the weld area adjacent to mating surfaces with clean scrapers.

NOTE

Do not use thumb as a scraping guide. Do not attempt to scrape on the back stroke.

3.1.5 Break the knife-like edge on the concave cut with a straight file.

3.1.6 Buff both surfaces to be welded with stainless steel wool.

3.1.7 After mechanical cleaning operations are complete, do not re-clean with a solvent.

3.1.8 To reduce the possibility of further contamination, wrap the scraped edges with aluminum foil and tape until the weld is ready to be made. Secure the tape in a manner that will not contaminate the weld zone.

4. CONTROL

4.1 Personnel shall insure that the following controls are exercised as applicable.

4.1.1 Clean solvents, wiping cloths, gloves, scrapers, files, and brushes are to be used.

4.1.2 All personnel involved in the cleaning operations shall wear clean gloves as required.

4.1.3 When cleaning operations begin, the cleaning cycles should be continuous when practical.

CLEANING ATM ALUMINUM COMPONENTS

1. SCOPE

1.1 Scope - This manufacturing procedure covers the requirements for precleaning, deoxidizing, conversion coating, anodizing, and final cleaning of ATM aluminum alloy components.

1.2 Applicability - Cleaning ATM aluminum components shall be accomplished by the procedures outlined in this procedure.

- Surfaces to be painted
- Surfaces to be conversion coated
- Surfaces to be anodized
- Surfaces to be final cleaned
- Surfaces to be recleaned

2. APPLICABLE DOCUMENTS

Federal

Federal Standard 209A - Clean Room and Work Station Requirements and Controlled Environment.

MSFC

MSFC Procedure 151 - Contamination Control and Environment Protection of Space Vehicles and Associated Equipment, Procedure for

MSFC-SPEC-164 - Cleanliness of Components for use in Oxygen, Fuel, and Pneumatic System

MSFC-STD-246A - Design and Operational Criteria of Controlled Environment Areas

MSFC 30M14500 - Apollo Telescope Mount Assembly

MSFC 50M02412 - ATM Cleanliness Specification

3. FACILITIES, EQUIPMENT, AND MATERIALS

3.1 Facility Cleanliness

3.1.1 Environmental Cleanliness levels required for packaging and cleaning, per 50M02412.

3.1.1.1. Class 30 000 Cleanroom - 30 000 particles per cubic foot, 0.5 microns and larger and 215 particles per cubic foot greater than 5.0 microns.

3.1.1.2 Class 100 000 Cleanroom - 100 000 particles per cubic foot, 0.5 microns and larger and 700 particles per cubic foot greater than 5 microns.

3.1.1.3 Relative humidity shall not exceed 50 percent at 72°F.

3.2 Equipment

3.2.1 Vacuum Oven

3.2.2 Heat Sealing Equipment

3.3 Materials and Chemicals

3.3.1 Acetone, Technical

3.3.2 Trichlorethylene, Types I and II

3.3.3 Trichlorotrifluoroethane (Freon, PCA)

3.3.4 Ethanol, Denatured

3.3.5 MEK

3.3.6 Turco 4215 Alkaline Cleaner

3.3.7 Turco Smut-go No. 1 equivalent

3.3.8 Iridite 14-2 (conversion coating)

3.3.9 Nylon-6 or equivalent, 2 to 4 mils (precleaned)

3.3.10 Polyethylene, 4 to 6 mils

3.3.11 Brown Kraft Paper

3.3.12 Nylon Gloves

3.4 Protection of Component

3.4.1 All components shall be wrapped with brown Kraft paper after cleaning in surface treat facilities.

3.4.2 All components shall be double bag wrapped after final cleaning in tube cleaning with inner bag being Nylon-6 and outer bag being polyethylene.

3.5 Cleanliness - The final cleaning cycles of all components shall be as specified in MSFC-SPEC-164.

4. CLEANING PROCEDURE FOR ALUMINUM COMPONENTS

4.1 Uncoated

4.1.1 Preclean - A cloth moistened with acetone shall be used in removing oil, ink, grease, and dirt from the surfaces.

4.1.2 Degrease - Parts shall be vapor degreased with trichloroethylene until vapors cease to condense on parts.

4.1.3 Dry - Parts shall be air dried until the trichloroethylene has vaporized.

4.1.4 Alkaline Clean - Parts shall be immersed in an agitating solution of Turco 4215 at 12 (± 3) oz/gal. at a temperature of 170°F ($\pm 5^\circ$) for 30 (± 5) minutes.

4.1.5 Rinse - Parts shall be rinsed in agitated tap water at 175°F ($\pm 5^\circ$) for 10 minutes minimum.

4.1.6 Deoxidize - Parts shall be immersed in an agitating solution of Turco Smut-go No. 1 at 12 (± 3) oz/gal. at ambient temperature for:

A1 6061 - 20 to 30 minutes

All others - 30 to 45 minutes

4.1.7 Rinse - Parts shall be rinsed in agitated demineralized water for 5 minutes minimum.

4.1.8 Dry - Parts shall be air dried or dried with missile grade air (handle with gloves).

4.1.9 Package - Parts shall be wrapped with brown Kraft paper before transferring to tube cleaning.

4.1.10 Final Cleaning - Parts shall be cleaned with Freon, PCA. See Paragraph 2.0, MSFC-SPEC-164, for particulate and NVR levels of cleanliness.

4.1.11 Dry - Parts shall be dried in vacuum oven at 25 to 27 inches of Mercury at 110°F ($\pm 10^\circ$) until dry (handle with gloves).

4.1.12 Package - Parts shall be double bag sealed with inner bag being Nylon-6 and outer bag being polyethylene.

4.2 Conversion Coating

4.2.1 Preclean - A cloth moistened with acetone shall be used in removing oil, ink, grease, and dirt from the surfaces.

4.2.2 Degrease - Parts shall be vapor degreased with trichloroethylene until vapors cease to condense on parts.

4.2.3 Dry - Parts shall be air dried until the trichloroethylene has vaporized.

4.2.4 Alkaline Clean - Parts shall be immersed in an agitating solution of Turco 4215 at 12 (± 3) oz/gal. at a temperature of 170°F ($\pm 5^\circ$) for 30 (± 5) minutes.

4.2.5 Rinse - Parts shall be rinsed in agitated tap water at 175°F (± 5) for 10 minutes minimum.

4.2.6 Deoxidize - Parts shall be immersed in an agitating solution of Turco Smutgo No. 1 at 12 (± 3) oz/gal. at ambient temperature for:

A1 6061 - 20 to 30 minutes

All others - 30 to 45 minutes

4.2.7 Rinse - Parts shall be rinsed in agitated demineralized water for 5 minutes minimum, with a pH of 6 to 8 at ambient temperature.

4.2.8 Conversion Coating - Parts shall be immersed in an agitating solution of Iridite 14-2 at 1.25 (± 0.25) oz/gal. at pH of 1.4 to 2.0 at ambient temperature for:

1 to 1 1/2 minutes for 2000 Series Aluminum
3 to 5 minutes for 6061 or 7075 Series Aluminum

4.2.9 Rinse - Parts shall be rinsed in agitated demineralized water for 5 minutes minimum.

4.2.10 Dry - Parts shall be air dried or dried with missile grade air (handle with gloves).

4.2.11 Package - Parts shall be wrapped with brown Kraft paper before transferring to Tube Cleaning.

4.2.12 Final Cleaning - Parts shall be cleaned with Freon, PCA. See Paragraph 2.0, MSFC-SPEC-164, for particulate and NVR levels of cleanliness.

4.2.13 Dry - Parts shall be dried in vacuum oven at 25 to 27 inches of Mercury at 110° F ($\pm 10^\circ$) until dry (handle with gloves).

4.2.14 Package - Parts shall be double bag sealed with inner bag being Nylon-6 and outer bag being polyethylene.

4.3 Aluminum to be Painted

4.3.1 Conversion Coated - Parts shall be cleaned with a cloth moistened with Acetone or MEK if the parts were received contaminated (dirt, grease, or oils).

4.3.2 Parts shall normally be cured for 12 hours after conversion coating.

4.4 Anodize Coating - (refers only to anodized parts).

4.4.1 Preclean - Parts shall be vapor degreased in trichloroethylene until vapors cease to condense on parts.

4.4.2 Dry - Parts shall be statically air dried or dried with hot missile grade air at 150 to 180° F.

4.4.3 Final Cleaning - Parts shall be cleaned with Freon for particulate matter (see Paragraph 2.0, MSFC-SPEC-164).

4.4.4 Dry - Parts shall be dried in vacuum oven at 25 to 27 inches of Mercury at 110° F ($\pm 10^\circ$) until dry (handle with gloves).

4.4.5 Package - Parts shall be double bag sealed with inner bag being Nylon-6 and outer bag being polyethylene.

4.5 Recleaning of Contaminated Aluminum Components - (Tube Cleaning Facilities).

4.5.1 Uncoated Aluminum

4.5.1.1 Preclean - A cloth moistened with Acetone or MEK shall be used to remove oil, ink, grease, and dirt from surface.

4.5.1.2 Degrease - Parts shall be vapor degreased with trichloroethylene until vapor ceases to condense on parts.

4.5.1.3 Dry - Parts shall be statically air dried or dried with hot missile grade air at 150 to 180° F.

4.5.1.4 Final Clean - Parts shall be cleaned with Freon (see Paragraph 2.0, MSFC-SPEC-164).

4.5.1.5 Dry - Parts shall be dried in vacuum oven at 25 to 27 inches of Mercury and temperature of 110° F ($\pm 10^\circ$) until dry (handle with gloves).

4.5.1.6 Package - Parts shall be double bag sealed with inner bag being Nylon-6 and outer bag being polyethylene.

4.5.1.7 Optional - Damaged parts requiring more than a Freon flush shall be recleaned in Surface Treat Facilities (see Paragraphs 4.1.2 through 4.1.12).

4.5.2 Conversion Coated

4.5.2.1 Preclean - A cloth moistened with Actone shall be used to remove any oil, dirt, grease, or ink from surfaces.

4.5.2.2 Degrease - Parts shall be vapor degreased with trichloroethylene until vapor ceases to condense on parts.

4.5.2.3 Dry - Parts shall be statically air dried or dried with hot missile grade air.

4.5.2.4 Final Cleaning - Parts shall be cleaned with Freon (see Paragraph 2.0, MSFC-SPEC-164 for particulate matter).

4.5.2.5 Dry - Parts shall be dried in vacuum oven at 25 to 27 inches of Mercury and temperature of 110°F ($\pm 10^\circ$) until dry (handle with gloves).

4.5.2.6 Package - Parts shall be double bag sealed with inner bag being Nylon-6 and outer bag being polyethylene.

4.5.2.7 Optional - Repeat Paragraphs 4.2 through 4.2.14 for damaged parts requiring more than hand reconversion coating.

4.5.3 Anodized Coating

4.5.3.1 Preclean - A cloth moistened with Acetone shall be used to remove ink, oil, dirt, and grease from surfaces.

4.5.3.2 Degrease - Parts shall be vapor degreased in trichloroethylene until vapors cease to condense on parts.

4.5.3.3 Dry - Parts shall be statically air dried or dried with hot missile grade air at 150 to 180°F (handle with gloves).

4.5.3.4 Final Clean - Parts shall be cleaned with Freon (see Paragraph 2.0, MSFC-SPEC-164, for particulate matter).

4.5.3.5 Dried - Parts shall be dried in vacuum oven at 25 to 27 inches of Mercury at 110°F ($\pm 10^\circ$) until dry (handle with gloves).

4.5.3.6 Package - Parts shall be double bag sealed with inner bag being Nylon-6 and outer bag being polyethylene.

5. QUALITY ASSURANCE

5.1 Cleanliness levels shall be maintained as specified in MSFC-SPEC-164.

5.2 Inspection Points

- 5.2.1 Paragraph 4.1.10.
- 5.2.2 Paragraph 4.2.9.
- 5.2.3 Paragraph 4.2.12.
- 5.2.4 Paragraph 4.4.3.
- 5.2.5 Paragraph 4.5.1.4.
- 5.2.6 Paragraph 4.5.2.4.
- 5.2.7 Paragraph 4.5.2.7.
- 5.2.8 Paragraph 4.5.3.4.

MANUFACTURING PROCESS DATA FOR
CLEANING LIQUID HYDROGEN (LH₂)
CRYOGENIC TANK FOR SPACE SHUTTLE PROTOTYPE I

1. SCOPE

This manufacturing process data describes a method for cleaning the conversion coating from the inside surface of the above described tank prior to shipping it to McDonnell Douglas, at Huntington Beach, California, for the internal installation of the insulation.

2. APPLICABLE DOCUMENTS

The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the latest revision shall apply.

SPECIFICATIONS

O-A-88 Acid, Nitric; Technical Grade

O-H-795 Hydrofluoric Acid, Technical

MSFC-PROC-404 Gases, Drying and Preservation, Cleanliness Level and Inspection Methods.

3. SAFETY

All applicable safety rules should be observed when preparing the chemical solutions, and when performing the actual cleaning operations.

Observe all standard safety requirements by wearing acid-proof clothing, boots, etc. Work shall be performed in a well ventilated area or while wearing air supply masks.

4. REQUIREMENTS

4.1 Facilities

4.1.1 The facilities located in the east end of test tower, Building 4707, will be used.

4.2 Equipment

4.2.1 Tanker truck with four each 1000 gallon tanks.

4.2.2 Tanker truck, 3500 gallon capacity for deionized water (stainless steel tank).

4.2.3 Rotating spray nozzles with raising and lowering capability.

4.2.4 Auxiliary solution tanks, pumping and control equipment.

4.2.5 Portable gasoline-driven pump.

4.2.6 Heating units for cleaning solutions and for missile grade air.

4.3 Materials

4.3.1 Non-silicated alkaline cleaner - Cee Bee MX-39.

4.3.2 Nitric Acid.

4.3.3 Hydrofluoric Acid.

4.3.4 Deionized Water.

4.3.5 Missile Grade Air.

5. PROCEDURES

NOTE

Maintain a continuous flow of missile grade air into the tank during these procedures.

5.1 Mount the tank vertically, with the retaining ring in position, in the east end of the hydrostatic test tower, Building 4707.

5.2 Degrease

5.2.1 Prepare solution using 6 (± 2) ounces of Cee Bee MX-39 per gallon deionized water and heat to 140 to 170° F.

5.2.2 Using the revolving spray nozzles mounted on the rotating arms, spray the entire interior surface of the tank with the above solution.

Maintain solution at 140 to 170° F during spraying operation.

Caution: Never allow the solution to fall below 4 percent Cee Bee MX-39 per gallon of deionized water.

5.3 Spray rinse with deionized water at 140 to 170° F and gradually reduce it to ambient temperatures.

5.4 Spray with a pickling solution of 20 percent HNO_3 , nitric acid, 0.5 percent HF, hydrofluoric acid, and deionized water at ambient temperature.

5.5 Spray with deionized water at ambient temperature.

5.6 Deoxidize with 20 percent HNO_3 nitric acid and deionized water by spraying the solution over the interior surface of the tank.

5.7 Final rinse with deionized water by thoroughly spraying the complete interior surface until there is no trace of the acid in the effluent water.

5.8 Dry the interior of the tank by blowing hot missile grade air into the tank.

6. CLOSURE OF TANK

After the tank is dry, seal the tank by attaching the dessicant breather unit to the access hole and attaching the cover plate to the aft opening. This must be done within 10 minutes after drying and removing the spray equipment.

MANUFACTURING PROCESS FOR CRYOGENIC FOAM
INSULATION APPLICATION TO S-1C/S-II TEST CONTAINER

1. SCOPE

This Manufacturing Process Data (MPD) details the approved Process Engineering Laboratory Procedures for application of the cryogenic insulation rigid polyurethane foams. One foam, Upjohn CPR-385-D, is applied to the S-1C portion, the other, Nopco-foam BX-250A, is applied to the S-II portion of the S-1C/S-II Test Container. Other MPD's pertinent to this MPD are:

- 1.1 MPD 25015 - Paint Removal from S-1C-S-II Test Container.
- 1.2 MPD 25017 - Cleaning of S-1C/S-II Test Container Prior to application of Spray Foam.
- 1.3 MPD 38001 - Process Control and Operating Procedures for Application of Nopco Foam BX-250A (applicable paragraphs to be used).
- 1.4 MPD 38003 - Process Control and Operating Procedures for Application of Upjohn CPR-385-D and CPR-368-2 Foams (applicable paragraphs to be used).
- 1.5 Spec. No. - Insulation Materials, Foam, Two-Pound Density, Spray Application to Test 10M01830. Articles, Spec. For. To be adhered to as close as possible.

2. MATERIALS

- 2.1 Nopco BX-250A components A (T) and B (R) in steel drums
- 2.2 Upjohn CPR-385-D (Spray Foam System) and Upjohn CPR-348 (Pour Foam System)
- 2.3 Adiprene L-100, or Narmco 7343 Resin
- 2.4 Moca or Narmco 7139 curing agent

- 2. 5 Methylene dianiline curing agent (MDA)
- 2. 6 Adhesive DS-1301
- 2. 7 Acetone
- 2. 8 Ethanol
- 2. 9 Methanol
- 2. 10 Freon, Type 113
- 2. 11 Methyl Ethyl Ketone
- 2. 12 Ethyl acetate, urethane grade
- 2. 13 Dow Corning Z-6020 primer
- 2. 14 Epocast primer "M," components B and C
- 2. 15 Ortho-phosphoric Acid, reagent grade
- 2. 16 Concentrated Nitric Acid, reagent grade
- 2. 17 Concentrated Sulfuric Acid, reagent grade
- 2. 18 Hydrofluorosilicic Acid, reagent grade
- 2. 19 Sodium Dichromate, reagent grade
- 2. 20 De-ionized water
- 2. 21 Cab-O-sil
- 2. 22 Petroleum Jelly
- 2. 23 Mylar Film, 5 mils thick, type 500 A
- 2. 24 Polyvinyl chloride or polyethylene film (5-10 mils)
- 2. 25 2-inch to 3-inch wide aluminum backed adhesive tape, 3M-425

- 2. 26 4-inch wide aluminum backed adhesive tape, 3M-428
- 2. 27 1-inch to 3-inch wide masking tape
- 2. 28 Teflon adhesive tape, 2-inch wide
- 2. 29 Waterproof adhesive tape, 4-inch wide
- 2. 30 Double backed adhesive tape, 2-inch wide
- 2. 31 Plater's tape, 2-inch to 3-inch wide, 3M-470
- 2. 32 36-inch wide brown paper
- 2. 33 Wet/dry sandpaper, 200 grit
- 2. 34 8 test panels 3/16 inch X 12 inch X 60 inch, 10 test panels
3/16 inch X 24 inch X 40 inch, 8 test panels 3/16 inch X 24 inch
X 60 inch
- 2. 35 5/8 inch diameter cork or rubber stoppers (tapered)
- 2. 36 Aluminum Alloy 7075-T6 0.020 inch X 12-inch
- 3. EQUIPMENT GENERAL
- 3. 1 Stationary access platform over bulkhead, PP-370-7654, modified
with support SF-14963
- 3. 2 33-foot diameter turntable, controllable velocity, AF-300-7016
- 3. 3 33-foot diameter holding fixture, HF-300-7074
- 3. 4 Vertically movable platform around HF-300-7074
- 3. 5 15-foot long curved drain pan, PF-01-2660
- 3. 6 Support fixture for 3 spray foam guns, SF-14856, with gun oscilla-
tor assembly SF-14856-1 and base SF-14856-2
- 3. 7 Sub-base adapter SF-14862 for SF-14856

- 3. 8 Foam machining fixture TMF-395-14845, 12-inch cutters
- 3. 9 Light tooling, foam barriers and plugs, MIT-14870
- 3. 10 Support fixture SF-14855 for TMF-14845
- 3. 11 Adapter TMF-395-14877 for TMF-395-14845
- 3. 12 Support fixture SF-14863 for TMF-395-14845
- 3. 13 Adapter beams MIT-14857 for PP-370-7654
- 3. 14 Sub-base support SF-14861 for SF-14855
- 3. 15 Angle barriers MIT-14864
- 3. 16 Machining fixture tilt adapter MIT-14945
- 3. 17 Guide rail MIT-14941 for PP-370-7654
- 3. 18 Individual gun oscillators MIT-14928
- 3. 19 Personnel platform PP-394-14875
- 3. 20 Step assembly PP-395-14667
- 3. 21 Fiberglas ladders with center support MIT-14960
- 3. 22 Access platform PP-14961 for primer
- 3. 23 Phenolic fibre and Fiberglas knives MIT-14972
- 3. 24 Personnel platform for sealer MIT-14967
- 3. 25 Molds for foam blanks MIT-14964
- 3. 26 Six Binks formulators, Type C Hot Hydraulic, with pressure pots
or bung pumps--three each for CPR-385 (1-to-1), three each of
Nopco BX-250-A (1-to-1.12)
- 3. 27 Six Binks airless spray guns, Model 43 P

- 3.28 Six Binks solvent supply pressure pots
- 3.29 Paint spray gun for the primers
- 3.30 Contact pyrometer
- 3.31 Compressed air (shop supply, 120 to 200 lb/in²)
- 3.32 Compressed nitrogen, dry, with pressure regulators
- 3.33 Plastic curtains around work area
- 3.34 Environmental control of temperature and humidity
- 3.35 Breathing air face masks
- 3.36 Protective clothing and gloves (plastic and cotton)
- 3.37 P. Q. V. equipment
- 3.38 Test equipment for determination of the foams density, tensile, and compressive strength
- 3.39 Infra-red heating lamps
- 3.40 Exhaust fan with hose
- 3.41 Nylon bristle paint brushes, 1/2-inch, 1-inch, and 2-inch wide

4. PROCEDURES

4.1 S-IC - General

4.1.1 Primer and Paint Removal

4.1.1.1 Remove zinc-chromate primer and paint from the bulkhead, the "Y" ring, and cylinder wall of the S-IC part, where required.

4.1.1.2 Remove paint and primer from the lower 24 inches of the upper skirt outside.

NOTE: These operations are accomplished in accordance with MPD No. 25015, "Paint Removal from S-IC/S-II Test Container."

4.2 Bulkhead - S-IC

4.2.1 Cleaning of the Bulkhead and the "Y" Ring

NOTE: Masking of the S-IC/S-II Test Container should begin below the bolt-ing ring and proceed upward in a shingle fashion.

4.2.1.1 After completion of structural modifications to Drawing No. 30M13976 and welding of the bottom edge of the S-IC unit to the upper edge of the S-II unit to Drawing No. 30M13975, install personnel platform No. PP-370-7654 modified to permit rotation of the entire test container under it. This fixture will be used for some of the subsequent work to be done on the bulkhead, along with platform PP-395-14875 and Fiberglas ladders.

4.2.1.2 Wipe the bulkhead and "Y" ring with MEK or acetone to remove any accumulation of dust and dirt resulting from prior work accomplished on the S-IC unit. As the solvent wiping proceeds, dry the surface with clean, lint-free cloth until no evidence of soil is visible on the clean wiper.

4.2.1.3 Mask off the upper four inches of the outer leg of the "Y" ring inside and out with a 4-inch wide waterproof tape. Make sure the fastener holes for the bar sections and the skin of forward skirt are clean as taping proceeds. (See Figure 4-1.)

4.2.1.4 Mask off with waterproof tape the lower 3 inches above and 3 inches below the S-IC/S-II horizontal weld line on the upper edge of the skin of the S-II unit. The circumferential 6-inch closeout need not be etched.

4.2.1.5 Protect with plastic sheeting and plater's tape the S-II unit sidewall where vinyl strippable coating was completely removed for welding and strain gage installations.

4.2.1.6 Using plater's tape, protect with plastic sheeting the vertical wall of the S-IC cylinder starting from the 4-inch tape on the "Y" ring to the 6-inch closeout at the joint of S-IC and S-II units.

4.2.1.7 Use 15-foot drain pan below outer leg of "Y" ring under plastic sheeting to collect effluent.

NOTE: This protection is required because cleaning and etching of the vertical wall will be done after completion of foaming of the bulkhead and installation of the forward skirt. It is preferable to use plater's tape wherever some degree of acidity may be present in the rinsing water due to etching.

4.2.1.8 Working on a limited area at a time, proceed to clean the bulkhead by etching, deoxidizing, rinsing, and air drying it in accordance with MPD-25017, observing all the specific safety precautions and utilizing fiberglass ladders with a center plywood support as an auxiliary tool.

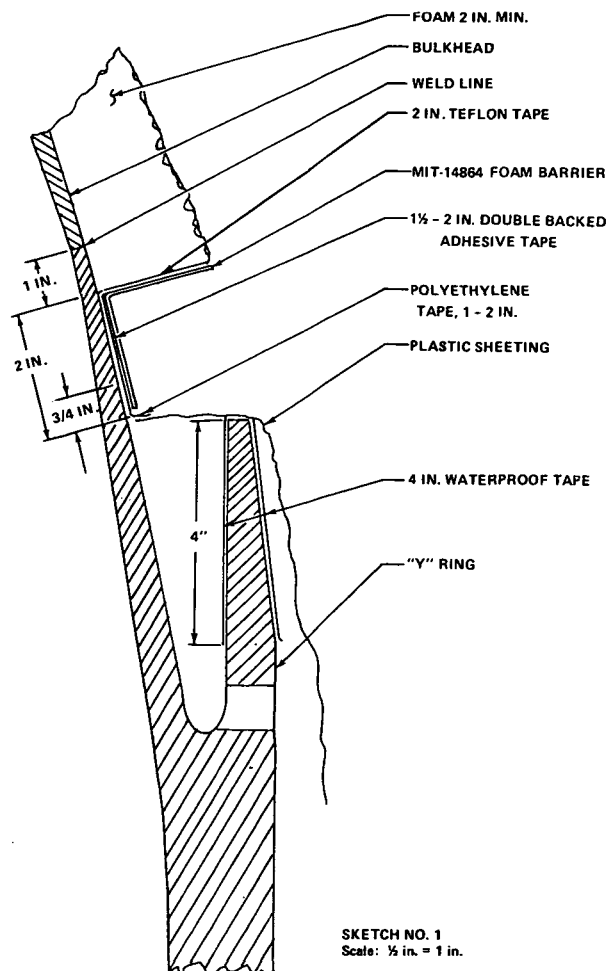


Figure 4-1. Bulkhead foam barrier and Y ring protection.

NOTE: There is no need to etch and rinse the inside surface of the "Y" ring as foaming of the bulkhead will not extend into it.

4. 2. 2 Priming the Bulkhead

4. 2. 2. 1 MPD 25017 requires water in the "Y" ring and polyethylene sheeting over it. Water can be drained now, but leave the sheeting on for protection of "Y" ring from foam overspray.

4. 2. 2. 2 Prepare the Z-6020 Dow-Corning primer diluted with ethanol (not methanol) and apply it to the cleaned and dried area of the bulkhead as specified in MPD-38003, Paragraphs 4.3.1 through 4.3.6, using a hand operated paint spray gun over a limited area while the bulkhead is being rotated by the turntable.

NOTE: Check that the surface is dry (2 hours minimum) before starting to prime.

4. 2. 2. 3 Continue priming each area until the entire surface of the bulkhead is primed. Allow the primer to cure at room temperature for a minimum of 2 hours.

4. 2. 3 Foam Barriers on the Bulkhead (Reference Drawing No. 30M1399I)

4. 2. 3. 1 To provide a foam barrier approximately 1 1/2 inches below the weld line of the bulkhead base segments to the inner leg of the "Y" ring, apply 1 1/2 to 2 inch wide double-backed adhesive tape just above the level of the outer leg of the "Y" ring. Continue all around the bulkhead. To this tape firmly apply flexible aluminum angle barrier MIT-14864, 2-inch by 2-inch. To the vertical leg of this barrier apply 2-inch wide teflon adhesive tape as a releasing agent. See Figure 4-1 and Section C-C of the referenced drawing.

NOTE: The double-backed tape can lap over the polyethylene tape holding the plastic sheeting required by MPD-25017. It may be necessary to apply wax to the metal surface before applying the double-backed tape; then remove the wax with a solvent after the tape is removed and reprime if a primer was there before the wax was applied.

4. 2. 3. 2 All other closeouts on the bulkhead called for in the referenced drawing in accordance with various section designations will be installed as specified by tooling provided for each section as follows:

Section:

Tool No.

B-B

MIT-14870

AH-AH (2 places)

AC-AC (2 places)

AG-AG (4 places)

AB-AE (1 place)

AF-AF (1 place)

4. 2. 3. 3 For Sections A1-AL and AM-AM use round wooden plugs 2-inches (10 places) and 3 inches (3 places) in diameter, respectively. Plugs to be hollowed out for the bolt head clearance have on top 3/8 inch dowels 3 inches long to indicate locations after spray foaming. Hold plugs in position by applying double-backed adhesive tape. Use Teflon Tape as the release agent for foam break away around the closeout tools.

4. 2. 3. 4 In all closeouts on the bulkhead where no foaming is specified by the drawing, use brown paper for protection of metal covers and bolts, held down by masking tape or double-backed adhesive.

NOTE: Access to the covers and bolts is necessary for leak testing.

4. 2. 4 Equipment Setup

4. 2. 4. 1 On the movable platform install material supplies, and on PP-370-7654 platform with guide rail MIT-14941, install two individual gun oscillators MIT-14928 as shown on Figure 4-2.

NOTE: All spray foaming variables that have been recently established and are being currently established shall be incorporated into a revision when the variables have all been established, or if needed earlier, at such time as the requirement is recognized. Sketches applicable to these variables will also be incorporated into the revision.

4. 2. 4. 2 For a specific gun set-up, see paragraph covering the specific operation.

4. 2. 4. 3 Connect shop air supply to the oscillating air motors and by adjusting the air flow control valves, verify the gun oscillating action. For the gun swing angle and other settings, see corresponding sketches.

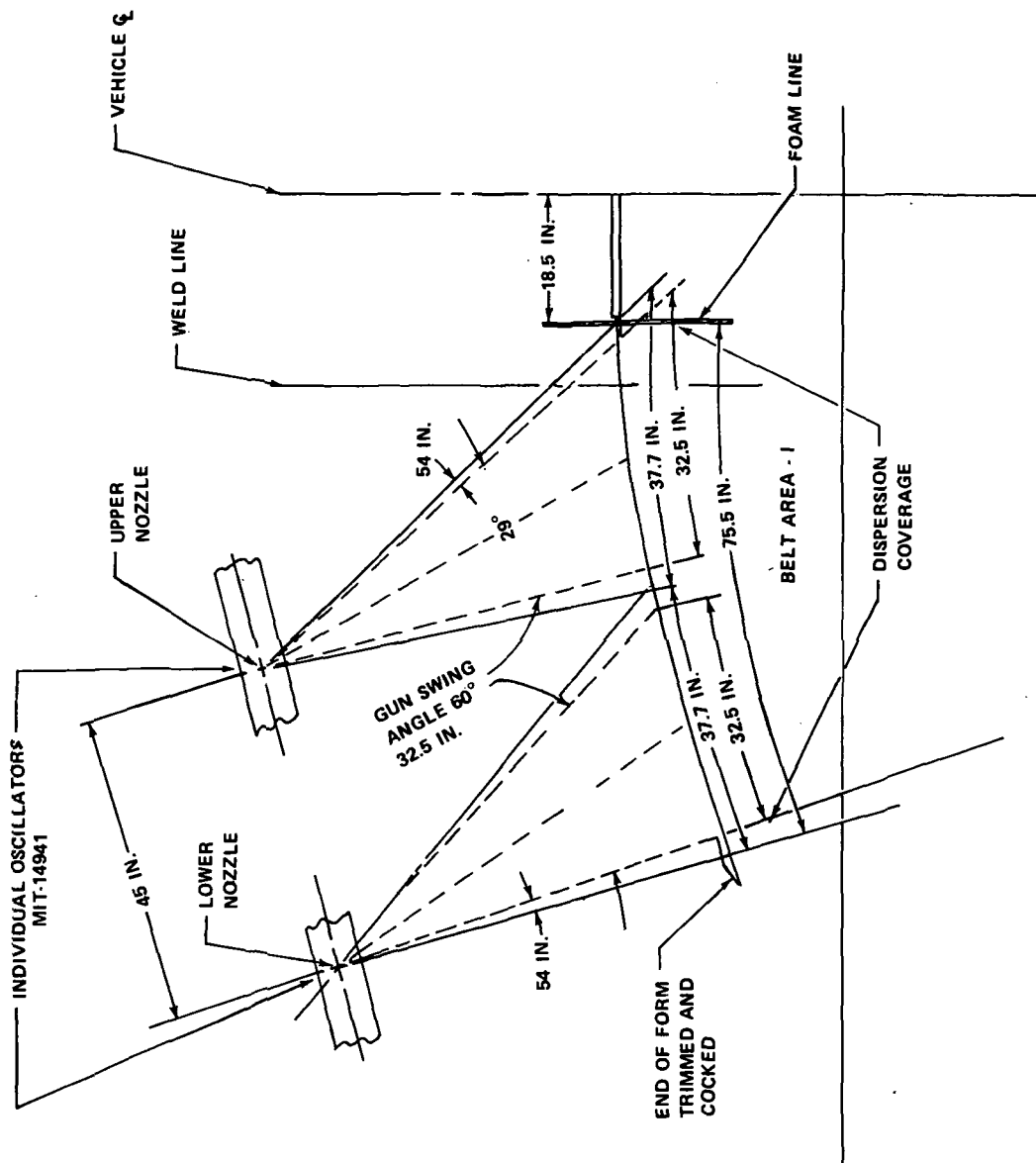


Figure 4-2. Gun FWD angle, belt area 2.

4. 2. 4. 4 Prepare two sets of the Upjohn Company CPR-385D spray foam components A and B, and connect them to the heaters of the Binks type "C" formulators. The method of connections can be standard either through the pressure pots or directly from the barrels with bung pumps.

NOTE: Both pairs of the components supply should be connected to the formulator in an identical manner.

4. 2. 4. 5 Fill the pressure pots and let two components stabilize at room temperature. Tighten "C" clamps of the lids and adjust nitrogen pressure to 110(±) 10 psi in both pots.

4. 2. 4. 6 Adjust the shop air pressure supply to the air motor driving stirring paddles so that the paddles do not turn in excess of 30 rpm.

4. 2. 4. 7 Connect the tracer hoses from the formulator outlets to the guns.

4. 2. 4. 8 Adjust the by-metal thermostat on top of the heaters so that the thermometers at the bottom of the heaters indicate 110° F(± 5°) for component A and 80° F (±5°) for component B.

4. 2. 4. 9 Set the tracer heat controls at 110° F.

NOTE: During the pre-heat time, occasionally trigger the gun to allow the components to flow through the heater past the thermometers, otherwise they will not read correctly.

4. 2. 4. 10 In case the bung pumps (Lark or Falcon) are used for material supply, the air pressure regulators of both pumps should be set at approximately 30 psi, resulting in 110 (±10) psi on both components. The N₂ blanket pressure should not exceed 3 to 4 psi.

4. 2. 4. 11 Before triggering the guns, prepare three solvent pressure pots by mixing one gallon of acetone with the same amount of methanol for each pot and through the solvent (black) noses connect two pots to the corresponding guns. (Keep the third pot as spare.)

4. 2. 4. 12 Connect the shop air supply to the solvent pot pressure regulator and adjust it to 30 to 40 psi; check that the solvent flows freely through the gun for each spray system.

4. 2. 4. 13 Purge the gun after each triggering. When the gun mounted on the oscillators, purging of the guns must be made into a waste basket.

4. 2. 5 Spray Foaming the Bulkhead, Belt Area No. 1

4. 2. 5. 1 Using the Upjohn Company CPR-385-D spray foam, check that components A and B and solvent pots are full, connections are not leaking, pressures and temperatures are set as specified in Section 4. 2. 4 of this MPD, and verify that the environmental conditions are within the limits of the Psychrometric Chart. With a contact pyrometer, check that the bulkhead surface temperature is 80° F (± 5°).

4. 2. 5. 2 Using one hand-held gun, check its action into a waste bucket and check foam density by the standard cup weight method, verify 1.8 to 2.3/ft³ density.

NOTE: Maintain and submit to R-QUAL in process data sheets covering items specified in paragraphs 4. 2. 5. 1 and 4. 2. 5. 2, and equipment operating parameters.

4. 2. 5. 3 The main access cover will be removed and sprayed separately with 3/4-inch foam thickness minimum, using No. 1520 nozzle .026-inch insert and .035-inch orifices.

4. 2. 5. 4 The remaining length of the arc of the bulkhead to be spray foamed is 226 inches, making the width of each of the 3 circumferential belt areas 75.3 inches. 2-gun position no. 1 is the belt area next to the main access cover. 2-gun position no. 2 is the belt area at the bottom of the bulkhead next to the "Y" ring. The 3d 2-gun position is the belt area between the first two.

4. 2. 5. 5 The 2 guns mounted on individual oscillators MIT-14928 with guide rail MIT-14941 on the modified personnel platform PP-370-7654 per paragraph 4. 2. 4. 1 are suitable for spraying belt areas No. 1 and No. 3. For belt area No. 2, support fixture SF-14856, modified, will be used due to interference with PP-370-7654.

NOTE: Since gun locations were determined empirically it is necessary to verify the foam distribution along the curve of the bulkhead intersection with a vertical plane, or a suitable test simulator.

4. 2. 5. 6 Apply aluminum foil to at least half of the circumference of the belt area no. 1, extending the paper well below and above the 75.3-inch belt to assure protection of the primed bulkhead surface from foam overspray.

NOTE: Approximately 1/4-inch thicker foam can be expected when spraying on aluminum foil than when spraying directly on the bulkhead due to the difference in heat sinks.

4. 2. 5. 7 Using nozzle no. 1520, orifice 0.035-inch for component "A"; 0.046-inch for component "B"; and insert 0.020-inch for the upper gun; nozzle No. 2620, orifice 0.035-inch for "A"; 0.046-inch for "B" and insert 0.026-inch for the lower gun. Test action of the guns into a waste bucket. Check output of each gun. Total output from the upper gun should be 3 lb/min., from the lower gun 4.8 lb/min.

NOTE: A 3-gun set-up is acceptable if said arrangement adequately covers area; location and arrangement to be shop determined.

4. 2. 5. 8 Adjust turn-table velocity to 16 feet per minute at 33 feet diameter. This will give 10 feet per minute to the lower edge Belt Area No. 1, which has 14.5 feet diameter.

NOTE: The above blanks will be filled after completion of simulator test program.

4. 2. 5. 9 Trigger both guns simultaneously and let the test container turn for 1 to 1 1/2 minutes, then stop.

4. 2. 5. 10 Observe foam distribution along the curve in the vertical plane and measure its depth at several points. It may be necessary to adjust the gun axis tilt or the swing angle or the nozzle distance from the surface.

4. 2. 5. 11 After adjustments are made, proceed with another trial run for 1 to 1 1/2 minutes and again check foam depth and distribution. If the distribution and depth of the foam are still unsatisfactory, a third trial may be required.

4. 2. 5. 12 When foam distribution and depth in various places are as good as can be obtained under the circumstances, remove the paper with foam and proceed to apply the foam directly to the bulkhead surface as follows.

4. 2. 5. 13 Mask off a portion of one belt area no. 1, marked "A" on Figure 4-3. Apply with masking tape sufficient amount of brown paper (marked P1) around Area "A" to protect bulkhead surface from over-spray as well as to permit start and stop of spray foaming on the left and right side of "A" (shaded area).

4. 2. 5. 14 After checking the 2 guns' action into a waste-bucket and starting on the left side of "A" at approximate turn-table velocity of 15 to 17 feet per minute, spray foam onto paper and slow down to 10 feet per minute upon reaching "A" to allow the required foam build-up, then speed up the velocity again for a few seconds to finish spraying the "A" on a paper. The intended circumferential profile of the foam is shown on Figure 4-3 as Step 1.

4. 2. 5. 15 Allow the sprayed foam to cure for a minimum of 4 hours then remove brown paper (P1) from the right and left sides of sprayed area "A" and trim these 2 edges of foam approximately 6 inches from the surface. There is no need to trim the upper edge at main access closeout at this time.

4. 2. 5. 16 Prepare Adiprene L-100 for brushing by adding to 100 parts of Adiprene by weight 12.5 parts by weight of Moca curing agent melted at 250° F(±10°) and mixing thoroughly. No degassing is necessary. Use 100 percent solids only, do not dilute with solvent.

NOTE: Selectron 5003 or Hetron 32A Polyester Resin prepared with 1/4 percent Cobalt and 1/2 percent MEKP may be used as Shop Alternate to Adiprene.

4. 2. 5. 17 Using stiff brush, 1 to 2 inches wide, apply Adiprene L-100 to the two side edges, and extend the application 3 to 4 inches onto untrimmed part of the foam. Protect metal surface from urethane application by strip of teflon tape applied adjacent to foam and extending approximately $1(\pm 1/8)$ inch onto foam edges. (This tape will be removed by peeling off after jelling has begun to prevent runs.)

NOTE: Use softer bristle brush for Polyester, tape is not required, coverage with Polyester is to tank wall.

4. 2. 5. 18 Allow Adiprene to cure for 12 hours minimum, then apply brown paper (marked P2) with masking tape on the left side of sprayed area to the trimmed edge. Starting from this brown paper apply another piece of brown paper (marked P3) extending from the left trimmed edge across the sprayed foam to the beginning of the trimmed edge on the right side of area "A." See step 2 on Figure 4-3.

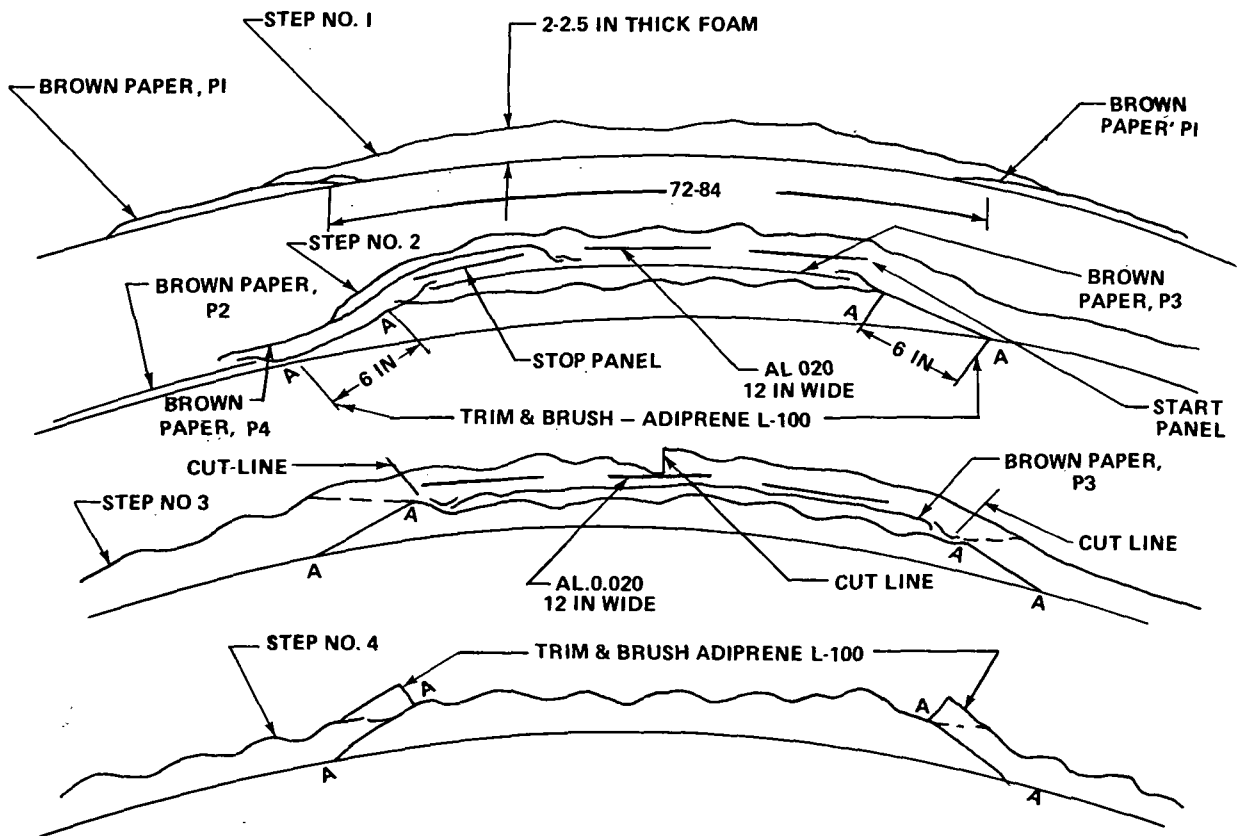
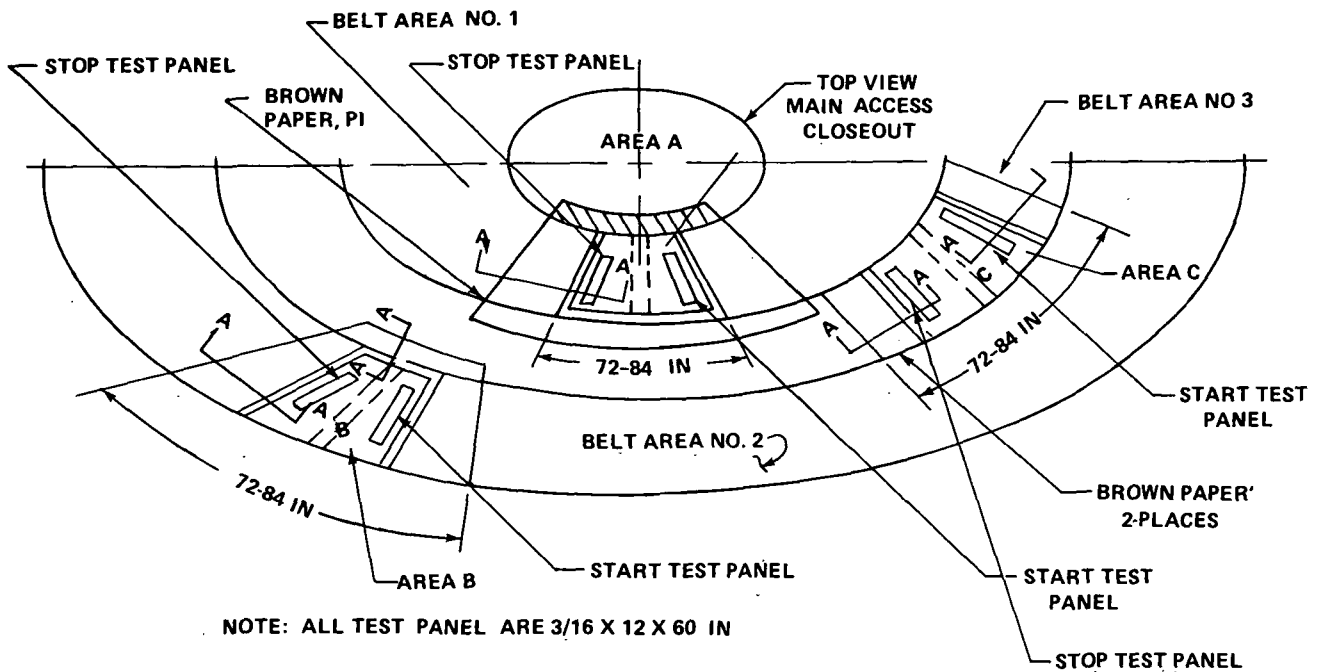


Figure 4-3. Bulkhead foaming belt area 1, 2, and 3.

NOTE: Polyester cure time is 1 hour minimum after application, 7 days maximum.

4. 2. 5. 19 Next to the right trimmed edge secure with masking tape two 3/16 x 20 a 24-inch aluminum alloy start test panels (one for each gun) over P3 brown paper. Next to the test panels apply a strip of approximately 0.020 inch thick, 12 inches wide aluminum alloy 7075-T6 extending from top to bottom of area "A." Next to the left trimmed edges secure two stop test panels of the same dimensions over the same brown paper marked P3.

NOTE: Test panels to be cleaned and primed same as tank surface.

4. 2. 5. 20 Apply another piece of brown paper marked P4 extending from top to bottom of area "A" and stopping at the left edge of the 0.020 strip, thus covering the stop test panels.

4. 2. 5. 21 After checking action of the guns into waste bucket, start spray foaming over the trimmed and resin coated right edge of area "A" foam and continue to go around belt area no. 1 (See step 2 of Figure 4-3.)

4. 2. 5. 22 Prior to reaching the left trimmed edge of the foam over area "A" remove P2 and P4 paper with foam and cut the P3 paper to the trimmed left edge of the foam and continue spraying over the surface of the stop test panels and stop at about the middle of the 0.020 aluminum strip. Allow the foam to cure a minimum of 4 hours then cut it with a knife right over the 0.020 aluminum strip. (See step 3 of Figure 4-3.)

4. 2. 5. 23 Cut the foam with a phenolic or fiberglass knife along the edges of the trimmed P3 paper and remove this paper along with the foam, 0.020 aluminum strip and start and stop test panels then cut the test panels out.

NOTE: All test panels are to be tested by R-ME-MW or as directed by R-QUAL.

4. 2. 5. 24 Trim the left-over excess of the foam as shown in step 4, Figure 4-3. Trim also the entire lower edge of belt area no. 1 in the manner used to trim the starting area "A" of this belt. Also trim the upper edge of this belt as shown in Section B-B, Drawing 30M13991. Then vacuum clean all the trimmed areas and apply Adiprene L-100 prepared and used as indicated in paragraphs 4. 2. 5. 16 and 4. 2. 5. 17. This includes vertical areas of the foam exposed by removal of closeout tool no. MIT-14870 around the main access cover.

NOTE: In this case, and all other cases on the entire S-IC/S-II Test Container where Adiprene L-100 is applied to any tapered closeout areas, the Adiprene should not touch the metal surface of the tank. A 1 ($\pm 1/8$) inch clearance between the edge of the Adiprene L-100 and the metal must be maintained on all tapered closeouts; all 90 degree closeouts require 1 ($\pm 1/16$) inch clearance.

4.2.6 Spray Foaming Bulkhead Areas Nos. 2 and 3 (See Figures 4-4 and 4-5).

4.2.6.1 Handling of starting area "B" of belt no. 2 and of starting area "C" of belt no. 3 is identical with that of belt no. 1 (including start and stop test panels) except, that trimming and preparation of the edges will be different because the area "B" in top edge will be trimmed in addition to the left and right side edges rather than bottom edge as it was in area "A" of belt No. 1. In area "C" only the left and right edges will be trimmed and prepared, but not the top nor bottom edges as the foam in this case will cover the prepared and resin coated edges of belt no. 1 and belt no. 2.

NOTE: There is no need to apply brown paper, P1 to the bottom edge of area "B" as there is a foam barrier and plastic sheeting below it as shown on Figure 4-1.

4.2.6.2 Gun locations and their swing angles are shown on Figures 4-4 and 4-5. For belt area no. 2, clean and assemble the lower gun with no. 3140 nozzle, 0.026-inch sapphire insert, and 0.035-inch orifice for component A and 0.046-inch orifice for component B. Assemble the upper gun with nozzle no. 2640, 0.026-inch insert, and 0.035-inch and 0.046-inch orifices.

For belt area no. 3, the nozzles and orifices are identical with belt area no. 2 for both guns and only inserts 0.020-inch for the lower gun and 0.026-inch for the upper gun should be used.

NOTE: Gun positions and angles are approximate and may be varied to achieve best results.

4.2.6.3 After spray foaming of entire belt area no. 2 all around the bulkhead is completed, allow the foam to cure for a minimum of 4 hours; then trim the upper edge of the belt area no. 2 and coat the edge with Adiprene L-100. Maintain 1 1/8-inch clearance between the lower edge of Adiprene and the metal surface. No Adiprene L-100 coating is required upon completion of foaming entire belt area no. 3.

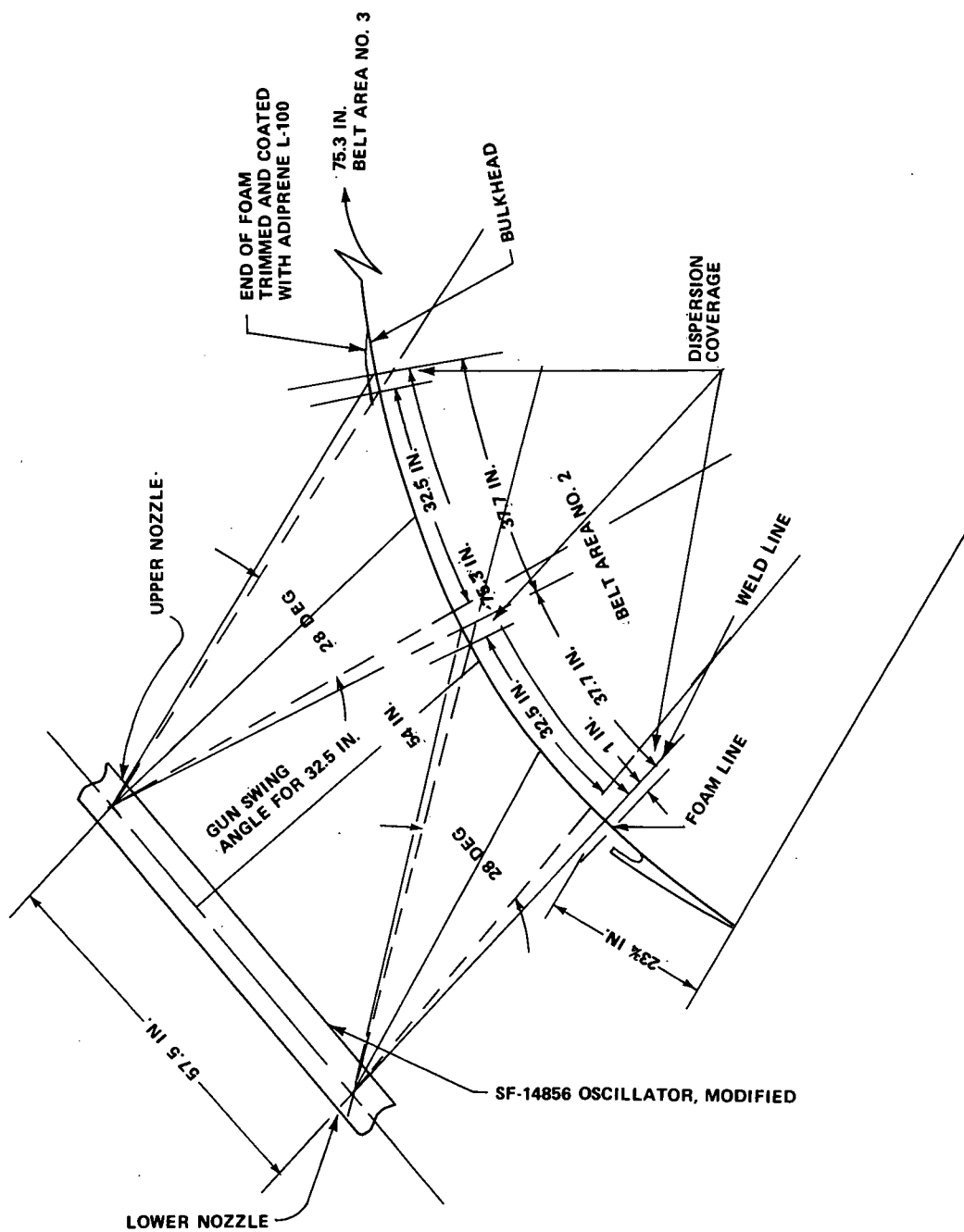


Figure 4-4. Gun swing angle belt area No. 2.

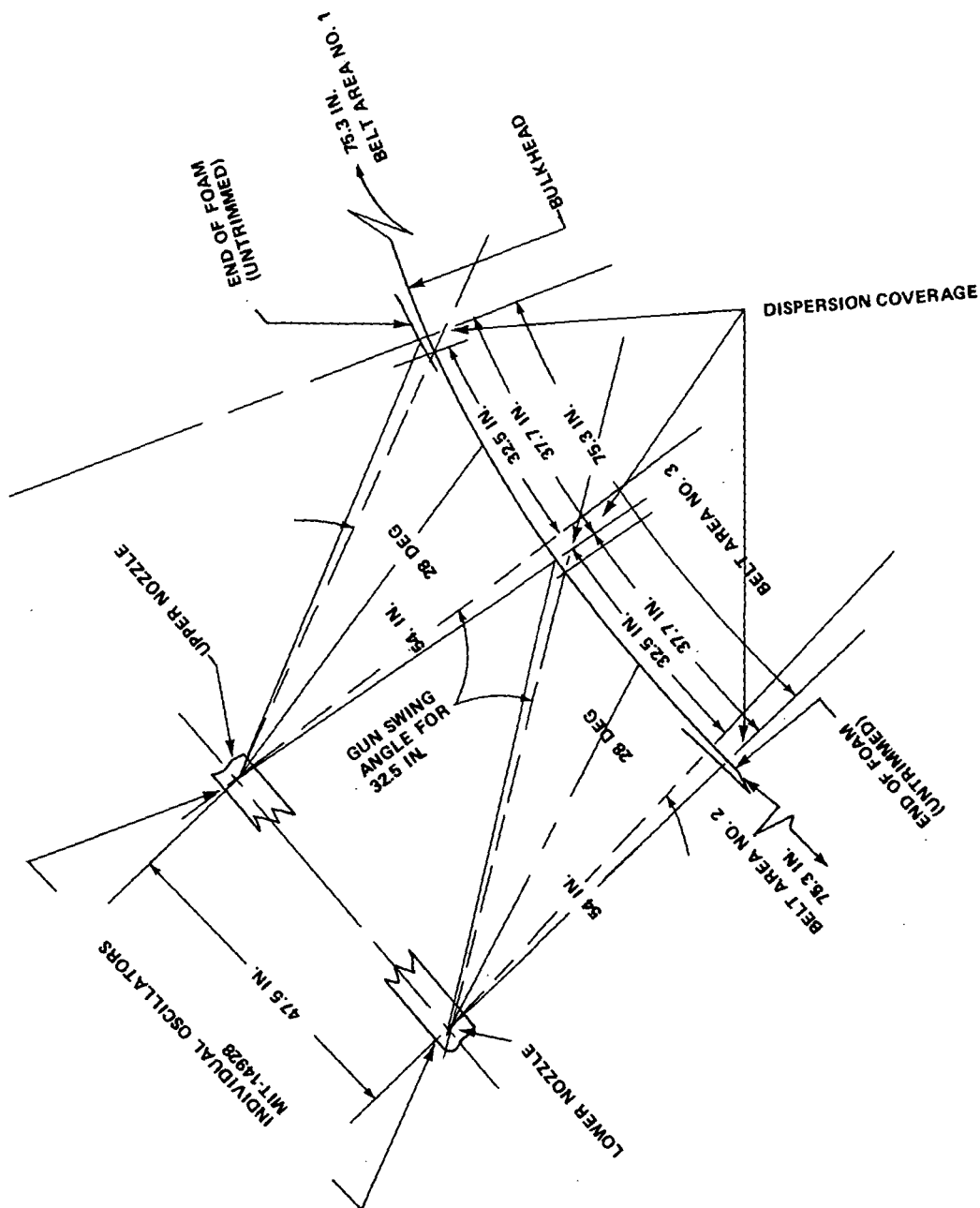


Figure 4-5. Gun swing angle belt area No. 3.

4. 2. 6. 4 Remove foam barrier MIT-14864 installed per paragraph 4. 2. 3. 1. Leave the plastic sheeting on, then take two machine cuts, 12 inches wide each, as shown in Section C-C, Drawing 30M13991, utilizing foam machining fixture TMP-395-14845 and tilt adapter MIT-14945.

4. 2. 6. 5 Remove all closeout tools that were installed in accordance with paragraphs 4. 2. 3. 2 and 4. 2. 3. 3. Leave the brown paper used for protection of metal covers and bolts per paragraph 4. 2. 3. 4 as is or reapply the paper if it has been removed.

4. 2. 6. 6 Using phenolic or fiberglass knife, cut a narrow "V" or "U" channel from the lowest point in all closeouts that have no mylar covers with a slope permitting drainage of water that otherwise would accumulate.

4. 2. 6. 7 As vacuuming proceeds, examine the cut and uncut foam surfaces for any discrepant areas (dents, holes, gouges, etc.) and repair these as detailed in paragraphs 4. 4. 5. 1 and 4. 4. 5. 2.

4. 2. 7 Outer Coating of Bulkhead

Method 1 (4. 2. 8) may be used for machined areas; Method 2 (4. 2. 9) will be used for remaining areas and as an outer coating over exposed Method 1 areas.

NOTE: Foam should be cured a minimum of 24 hours prior to application of outer coating.

4. 2. 8 Adiprene L-100/Moca Cure (Brushing)

4. 2. 8. 1 As in paragraph 4. 2. 5. 16 prepare Adiprene L-100 for brushing by mixing by weight 100 parts of Adiprene L-100 with 12.5 parts by weight of Moca curing agent melted at 250° F(±10°). Mix thoroughly at room temperature. No degassing is necessary.

4. 2. 8. 2 Apply a uniform coat of this compound over all machined bulkhead areas and allow to cure for 12 to 24 hours at atmospheric pressure and room temperature (65 to 90° F).

NOTE: 100 percent solid Adiprene L-100/Moca will be used on all machined surfaces. This will not be diluted with solvents. The 100 percent adhesive can be applied to a cut foam surface 4 hours after spray foaming.

4. 2. 9 Adiprene L-100/Methylene Dianiline (MDA, Spraying)

4. 2. 9. 1 Using a cleaned Binks Formulator Type "C" (1-to-1 ratio) but without heaters, mix by weight in one pressure pot at room temperature 650 parts of Adiprene L-100 and 348 parts by weight of ethyl acetate.

4. 2. 9. 2 In the other pressure pot, dissolve 62.4 parts by weight of Methylene Dianiline (MDA) curing agent in 853 parts by weight of urethane grade ethyl acetate.

4. 2. 9. 3 Prepare Binks Solvent pressure pot, filling it with the necessary amount of ethyl acetate, urethane grade.

4. 2. 9. 4 Prepare a cleaned Binks airless spray gun, Model 43P, with No. 9-1820 nozzle, 0.020-inch Sapphire insert, and two 0.035-inch orifices.

4. 2. 9. 5 By-passing the formulator heaters, connect the pressure pots, formulator, solvent pressure pot, and gun specified hoses. No heat for the tracer hoses is required.

4. 2. 9. 6 Tighten "C" clamps of the lids and adjust dry nitrogen pressure of both pots to 110 (± 10) psi.

4. 2. 9. 7 Adjust the shop air supply to the air motor driving stirring paddles so that they do not turn in excess of 30 rpm.

4. 2. 9. 8 Connect the shop air supply to the solvent pot pressure regulator and adjust it to 30 to 40 psi. Check that the solvent flows freely through the gun.

NOTE: With the formulator and materials supply located on the movable platform, the 25-foot length tracer hoses should be sufficient to reach any place on the bulkhead from the top of personnel platform, PP-370-7654. Modified, however, the 50-foot long hose should be used to eliminate changing hoses after spray foaming has commenced.

4. 2. 9. 9 With the test container turning at suitable velocity under the personnel platform, after testing the gun action into a waste bucket, proceed to apply the outer coating to all the foamed bulkhead surfaces while oscillating the gun by hand and holding it 24 to 36 inches from the surface. Testing the gun action is necessary to assure that the solvent has been flushed from the gun.

4. 2. 9. 10 The desired outer coating thickness is 10 to 15 mils, while the dry film build-up is about 5 mils per pass; consequently, a minimum of two passes are needed.

4. 2. 9. 11 For complete cure of the outer coating, 7 days are required at room temperature.

4. 2. 9. 12 Upon completion of the outer coating application, the plastic sheeting can be removed from the "Y" ring along with the two strips of 4-inch waterproof tape from the outer leg of the ring.

4. 2. 10 Main Access Closeout

4. 2. 10. 1 After outer coating has cured for 12 to 24 hours, remove from all bulkhead closeouts the brown paper used per paragraph 4. 2. 3. 4 for protection of the metal covers and bolts.

4. 2. 10. 2 From a sheet of 5 mils thick Mylar type 500A cleaned with either MEK or acetone, cut out one each of the following circular strips:

No. 1. 42 inches o.d. and 37 inches i.d.

No. 2. 39 inches o.d. and 27 inches i.d.

No. 3. 29 inches o.d. and 24 inches i.d.

4. 2. 10. 3 Prepare Adiprene L-100/Moca adhesive compound as in paragraph 4. 2. 8. 1, then, using a stiff brush, apply a thin coat of the compound to one face of Circle No. 1 and Circle No. 3.

NOTE: Wear clean cotton gloves during the Mylar cutting and bonding operation.

4. 2. 10. 4 Apply Circle No. 1 and Circle No. 3 with the wet side down to the coated foam around main access and its cover as indicated in Section B-B, Drawing 30M13991. With a small hand roller, apply light pressure to Mylar and smooth out wrinkles and bubbles, if any. Let cure overnight.

NOTE: Roller pressure should not crush or dent the foam.

4. 2. 10. 5 Meanwhile, punch a 0.38-inch diameter hole in one location of Circle No. 2 midway between its inner and outer edges; and on one face apply Adiprene L-100 compound around the hole in 2 to 2 1/2 inch diameter.

4. 2. 10. 6 Clean the bonding face of purge fitting 30M13993 with acetone or MEK and apply to that face a light coat of the Adiprene L-100 compound. Then, with a light pressure apply the coated face of the purge fitting to the coated spot of Circle No. 2. Make sure that the hole in Mylar and the hole in purge fitting are aligned and unobstructed by the Adiprene L-100 compound. Let cure overnight undisturbed.

4. 2. 10. 7 Apply Circle No. 2 to both Circles No. 1 and No. 3 and again roll out any wrinkles or bubbles from the bond area.

4. 2. 10. 8 Apply 2-inch wide aluminum backed adhesive tape in two circles over the inner and outer edges of Mylar Circle No. 2. One piece of aluminum tape should have 41 inches o.d. and 37 inches i.d. The other piece should have 29 inches o.d. and 25 inches i.d.

NOTE: The hand roller will help to produce a firm contact between the aluminum tape and Mylar, but it may be necessary to snip the inner edges of both aluminum tape strips to avoid wrinkles by allowing the tape tabs to lay over each other.

4. 3 Sidewall - S-IC

4. 3. 1 Cleaning and Surface Preparation of the Forward Skirt and S-IC Sidewall

4. 3. 1. 1 After completion of installation and attachment of the forward skirt to the Y-ring, final cleaning operations will begin on the forward skirt and S-IC sidewall by first covering the 16 weep holes with waterproof tape.

4. 3. 1. 2 Lightly sand the lower 2-feet of the forward skirt. Wipe the skirt, "Y" ring, and S-IC sidewall with MEK or acetone to remove any accumulation of dust and dirt resulting from prior work accomplished on the S-IC unit. As the solvent wiping proceeds, dry the surface with a clean, lint-free cloth until no evidence of soil is visible on the clean wiper.

4. 3. 1. 3 Protect the lower 24 inches of the forward skirt with hat sections from acid etching and rinsing by using plastic sheeting and waterproof tape.

4. 3. 1. 4 Mask off with waterproof tape the lower 3 inches above and 3 inches below the S-IC/S-II horizontal weld line on the upper edge of the skin of the S-II unit. The circumferential 6-inch closeout need not be etched.

4.3.1.5 To work on an approximately 10-foot by 10-foot area, secure the 15-foot drain pan at the 6-inch circumferential closeout and using plater's tape, apply a 10-inch wide by 15-foot long strip of polyethylene or polyvinyl chloride at the lower edge of the working area so that the untaped side is inside the pan.

4.3.1.6 Using the waterproof tape, protect with plastic sheeting the sidewall of the S-II unit below the 6-inch closeout at the joint of S-IC and S-II units.

NOTE: This protection is needed because cleaning and etching of the S-II sidewall will be done after completion of cleaning and foaming of the S-IC sidewall.

4.3.1.7 Work on a limited area at a time, and proceed to clean the sidewall by etching, deoxydizing, rinsing, and air drying it in accordance with MPD-25017, observing all the specified safety precautions.

4.3.2 Priming the Sidewall

4.3.2.1 Remove plastic sheeting and tape from the lower 24 inches of the forward skirt.

4.3.2.2 Prepare the Z-6020 Dow-Corning primer diluted with ethanol (not methanol) and apply it to the cleaned and dried area of the forward skirt and S-IC sidewall areas as specified in MPD-38003, paragraphs 4.3.1 through 4.3.6 by using a hand-operated paint spray gun over a limited area while the sidewall is being rotated by the turn-table.

NOTE: To insure that the surface is dry, wait a minimum of 2 hours after rinsing and air drying before starting to prime. Primed surfaces shall be spray foamed within 7 days after curing of the Z-6020 primer.

4.3.2.3 Continue priming each area until the entire surface of the forward skirt (lower 2 feet) and S-IC sidewall have been primed. Allow the primer to cure at room temperature for a minimum of 2 hours.

4.3.3 Foam Area Preparation

4.3.3.1 Remove waterproof tape and insert 16 drain tubes 30M13996 into the weep holes of the "Y" ring and plug them with 5/8 inch diameter tapered rubber or cork stoppers.

4.3.3.2 Insert wooden rectangular plugs into bottom ends of the hat sections of the skirt.

4.3.3.3 Protect the "Y" ring and the forward skirt from over spray by masking with brown paper the area above the "Y" ring/sidewall weld line.

4.3.3.4 Prepare the closeout of S-IC/S-II weld line (station 425) as shown on Figure 4-6 with both angles at 90 degrees (MIT-14864-1).

NOTE: The two areas will be spray foamed independently, i.e., the area of forward skirt/"Y" ring will be spray foamed using one setup and the area between the "Y" ring and station 425 closeout will be spray foamed using another setup.

4.3.4 Equipment Setup - S-IC Area

4.3.4.1 Install sub-base SF-14862 on the movable platform to adapt the tool No. SF-14856 with oscillating 3-gun arrangement to the platform. Have the material supplies also on the movable platform.

NOTE: Locate sub-base SF-14862 on the platform so that the impinging stream from the guns will be in the radial direction with respect to the tank surface and the gun nozzle will be approximately 54 inches from it.

4.3.4.2 Install two spray guns in oscillator SF-14856. Connect shop air supply to the oscillating and skate air motors and by adjusting the air flow control valves, verify the gun oscillating action and up and down motion of the skate. For the gun swing angle and locations, see corresponding Figures 4-7 and 4-8.

4.3.4.3 Both guns for the sidewall area should be assembled with cleaned No. 3140 nozzles, 0.026-inch sapphire inserts and 0.035-inch orifices for component A and 0.046-inch for component B. For the forward skirt/"Y" ring area, use nozzle No. 3140 with .026-inch insert and .035-inch orifice for component A and .046-inch orifice for component B.

4.3.4.4 Prepare two sets of Upjohn Company CPR-385D spray foam components A and B, and connect them to the heaters of the Binks Type "C" formulators. The method of connections can be standard either through the pressure pots or directly from the barrels with bung pumps. (Keep the third set as spare.)

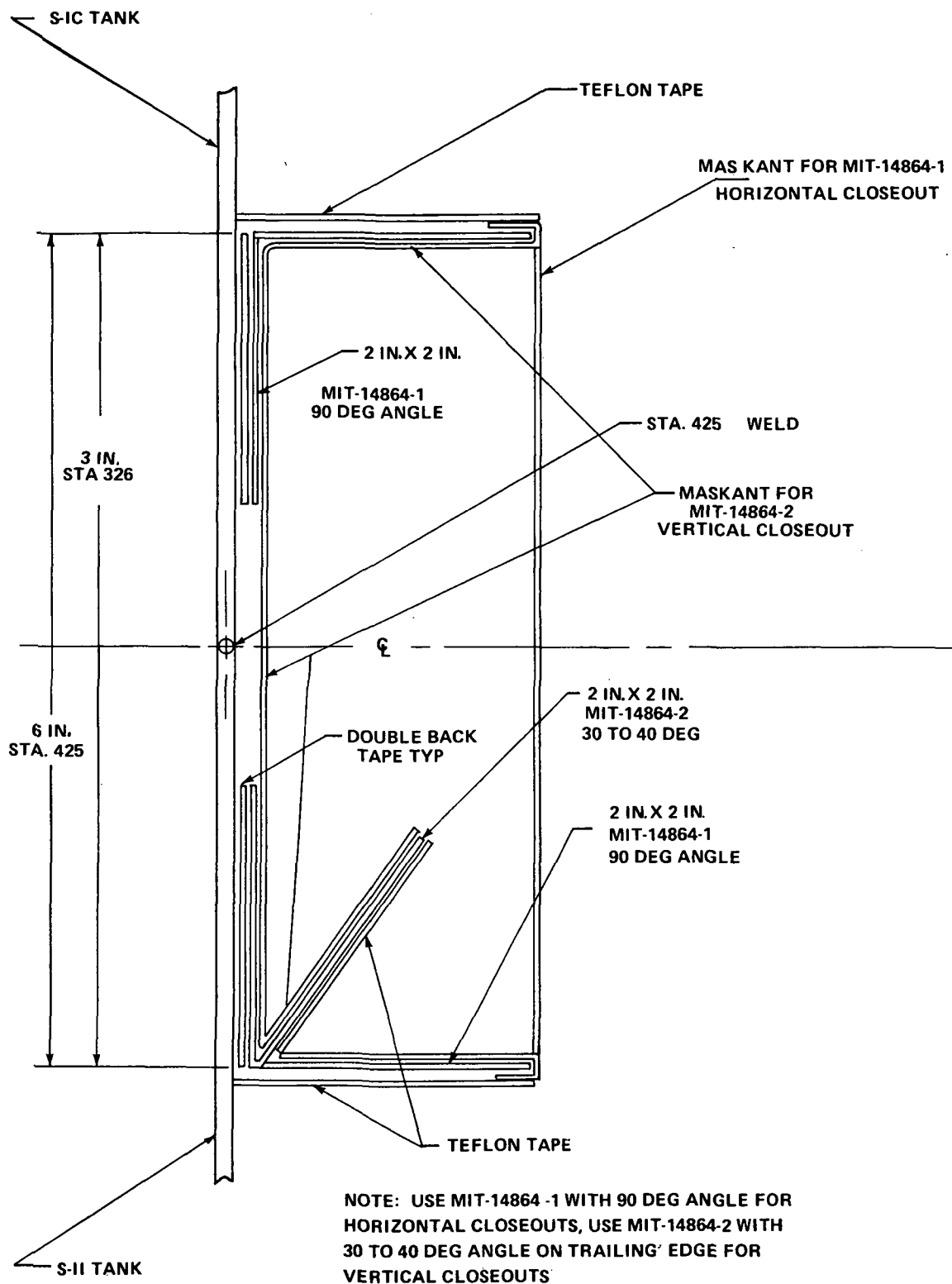


Figure 4-6. Foam edge weld closeout, sta 425 and sta 326.

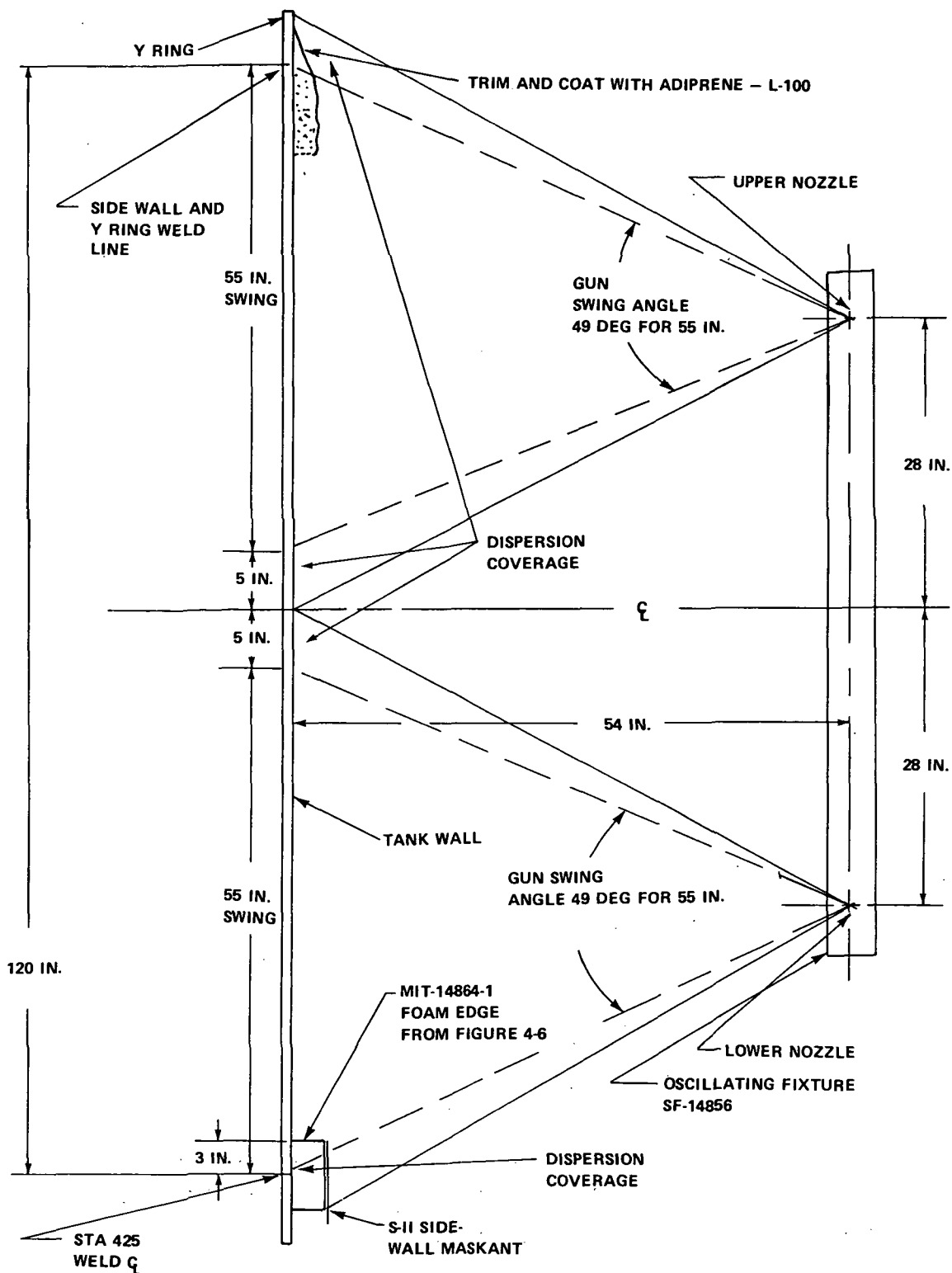


Figure 4-7. S-IC sidewall gun swing angle.

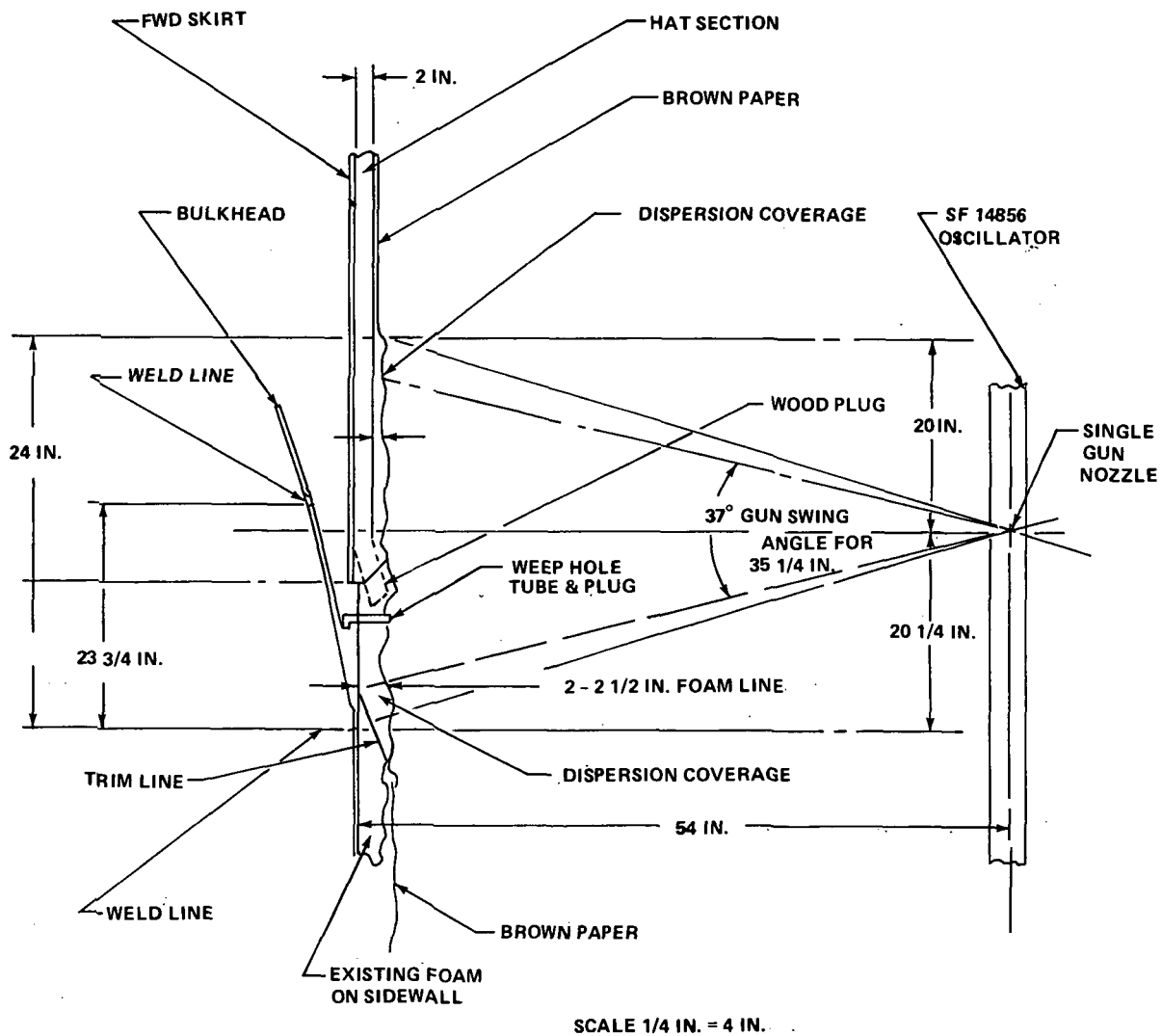


Figure 4-8. FWD skirt/Y ring gun swing angle.

NOTE: Both pairs of the components supply should be connected to the formulators in an identical manner.

4. 3. 4. 5 Fill the pressure pots and let two components stabilize at room temperature. Tighten "C" clamps of the lids and adjust nitrogen pressure to 110 (± 10) psi in both pots.

4. 3. 4. 6 Adjust the shop air pressure supply to the air motor driving stirring paddles so that the paddles do not turn in excess of 30 rpm.

4. 3. 4. 7 Connect the tracer hoses from the formulator outlets to the guns and set the tracer heat controls at 110° F.

4. 3. 4. 8 Adjust the by-metal thermostat on top of the heaters so that the thermometers at the bottom of the heaters indicate 110° F ($\pm 5^\circ$) for component A and 80° F ($\pm 5^\circ$) for component B.

4. 3. 4. 9 During the pre-heat time, occasionally trigger the gun to allow the components to flow through the heater past the thermometers, otherwise they will not read correctly.

4. 3. 4. 10 In case the bung pumps (Lark or Falcon) are used for material supply, the air pressure regulators of both pumps should be set at approximately 30 psi, resulting in 110 (± 10) psi on both components. N₂ blanket pressure should not exceed 3 to 4 psi.

4. 3. 4. 11 Before triggering the guns, prepare three solvent pressure pots by mixing 1 gallon of acetone with the same amount of methanol for each pot, and through the solvent (black) hoses connect two pots to the corresponding guns. (Keep the third pot as spare.)

4. 3. 4. 12 Connect the shop air supply to the solvent pot pressure regulator and adjust it to 30 to 40 psi, check that the solvent flows freely through the gun.

4. 3. 4. 13 Purge the gun after each triggering. With guns mounted on oscillator SF-14856, purging of the guns must be made into a waste bucket.

4. 3. 5 Spray Foam Application

4.3.5.1 Using the Upjohn Company CPR-385-D spray foam, check that components A and B and solvent pots are full, connections are not leaking, pressures and temperatures are set as specified in Section 4.3.4 of this MPD, and verify that the environmental conditions are within the limits of the Psychrometric Chart. With a contact pyrometer check that the sidewall surface temperature is 80° F(±5°).

4.3.5.2 Check action of the guns into a waste bucket and verify 1.8 to 2.3 lbs/ft³ density of foam by the standard cup weight method.

NOTE: Maintain and submit to R-QUAL in-process data sheets covering items specified in paragraphs 4.3.5.1 and 4.3.5.2 and equipment operating parameters.

4.3.5.3 Expose an 8- to 10-foot wide portion of the S-IC sidewall and prepare for start and stop panels as was done in bulkhead area "A", belt No. 1.

4.3.5.4 Using gun set-up as outlined on Figure 4-7, spray foam the exposed portion of the S-IC sidewall as follows:

- Actuate oscillator and spray gun with turntable rotating and apply 1/4 inch of foam (minimum).
- As exposed portion of the S-IC sidewall rotates before gun, reduce rotational speed to apply 2 inches of foam (minimum).
- As the end of exposed portion of the S-IC sidewall approaches, increase rotational speed to again apply 1/4-inch (minimum) of foam.
- After exposed portion of the S-IC sidewall has been foamed, de-actuate and flush spray gun and stop rotation of turntable, do not allow solvent to touch foam.
- After a 4-hour (minimum) delay remove maskant at each end of the foamed area and sand exposed vertical edges of foam to produce 6-inch wide taper using 200-grit abrasive paper for final sanding.
- Coat sanded surfaces with a 0.010 to 0.020-inch thick layer of Narmco 7343/7139 or Adiprene L-100/Moca Compound and allow to cure for 12 hours minimum, 24 hours maximum. Maintain 1 (±1/8)-inch clearance between Adiprene and metal surface.

NOTE: Selectron 5003 or Hetron 32A Polyester may be substituted for Adiprene, see paragraphs 4.2.5.16, 4.2.5.17, and 4.2.5.18.

4.3.5.5 Apply four start and stop panels in a sequence and manner similar to the method used on bulkhead belt areas. Using setup as shown on Figure 4-7, proceed to spray foam the balance of the S-IC sidewall circumference including the four test panels. Let foam cure for 4 hours minimum.

4.3.5.6 Remove brown paper with foam and cut out the test panels, using a fiberglass knife.

NOTE: All test panels are to be tested by R-ME-MW or as directed by R-QUAL.

4.3.5.7 After 4-hour (minimum) delay, sand the top edge of the foam to produce 6 inch wide taper using 200-grit abrasive paper for final sanding.

4.3.5.8 Coat sanded surfaces with 0.010 to 0.020 inch thick layer of Narmco 7343/7139 or Adiprene L-100/Moca Compound and allow to cure for 12 hours minimum to 24 hours maximum. Maintain 1 (\pm 1/8) -inch clearance between Adiprene and the metal surface.

4.3.5.9 Expose a 8- to 10-foot wide portion of the forward skirt/"Y" Ring and prepare for start and stop panels as was done in bulkhead area "A", belt No. 1.

4.3.5.10 Using gun setup as outlined on Figure 4-8, spray foam the exposed portion of the forward skirt and "Y" ring as outlined in paragraph 4.3.5.4.

NOTE: The two gun setup is acceptable if it adequately covers the area. Gun locations and arrangement will be shop determined.

4.3.5.11 Apply one start and one stop panel and spray foam the balance of the forward skirt and "Y" ring circumferences. There should be a minimum of 1/2 to 3/4 inch foam thickness over the hat sections. Let foam cure for 4 hours minimum.

4.3.5.12 Remove the brown paper with foam and cut out the test panels, using a plastic knife.

NOTE: All test panels are to be tested by R-ME-MW or as directed by R-QUAL.

4.3.5.13 Remove wooden plugs from the hat sections of the forward skirt and the plugs from 16 "weep" hole drain tubes.

4.3.5.14 Trim the foam just below the hat section with approximately 10 to 15 degrees downward slope to allow for drainage of water.

4.3.5.15 Waterproof the trimmed foam surface by coating it with 0.010 to 0.020-inch thick layer of Narmco 7343/7139 or Adiprene L 100/Moca Compound.

4.3.6 Outer Coating - S-IC Sidewall

4.3.6.1 Coating application to be same as for Forward Bulkhead, see paragraph 4.2.7.1 and 4.2.7.2.

4.4 S-II Portion Sidewall Spray Foaming (Stations 425 to 283)

4.4.1 Surface Preparation

4.4.1.1 Work on an area approximately 8 by 10 feet along the circumference of the tank at a time. Secure the 15-foot long drain pan at the bottom edge of the area to be cleaned after removal of protective plastic sheeting from stations 425 to 326 (weld line of cylinders no. 1 and no. 2).

4.4.1.2 Using plater's tape, apply a strip of polyvinyl chloride or polyethylene, approximately 10 inches wide by 15 feet long at the lower edge of the area so that the untaped side of the strip is inside the drain pan.

NOTE: Use of plater's tape is preferred whenever some degree of acidity may be present in the rinsing water due to etching.

4.4.1.3 Protect the protrusions of flanges and bolted covers from acid etching and rinsing with plastic sheeting, and waterproof tape the following sections: (drawing no. 30M13991) K-K, 2 places; T-T, 5 places; V-V, 1 place; Y-Y, 5 places; and AA-AA, 1 place.

4.4.1.4 Protect all the blind topped holes and Helicoil inserts with 1/4-28 to 3/8-24 hex head bolts, approximately 1/2 inch long with sealing washers (Neoprene or Nylon).

4.4.1.5 Protect with plastic sheeting and waterproof tape cylinder no. 2 with stringers and the bolting ring. Let the sheeting extend well below Station 283 to prevent effluent from getting into the hat sections of the aft skirt or damaging the strain gages that are installed on it.

4. 4. 1. 6 Proceed to prepare the acid etch paste and de-oxidizer paste as specified in MPD-38001, "Process Control and Operating Procedure for Application of Nopco Foam BX-250A Urethane Foam Insulation", paragraphs 4. 2. 1a and b.

4. 4. 1. 7 Following MPD-25017, "Cleaning of S-IC/S-II Test Container Prior to Application of Spray Foam", as a guide and working on a limited area at a time, apply the acid etchant and the deoxidizer, rinse, then dry with hot or forced air and protect the cleaned surface as indicated in MPD-38001, paragraphs 4. 2. 2, 4. 2. 3, and 4. 2. 5.

4. 4. 1. 8 Repeat the etching and de-oxidizing procedure on each area until the entire cylinder no. 1 surface on the S-II unit is prepared for priming, including aluminum adhesive tape protecting strain gages and their leads.

NOTE: Take care to avoid damage to strain gages already in place.

4. 4. 1. 9 After the surface above Station 326 has been cleaned and dried, remove plastic sheeting below Station 326 and wipe thoroughly with acetone or MEK cylinder no. 2, the stringers, the bolting ring, and bolts to prepare these surfaces for priming.

4. 4. 2 Priming S-II Part

4. 4. 2. 1 Rotate the tank 10 feet so that the next 8 by 10-foot area can be worked on while the first cleaned area is being primed.

4. 4. 2. 2 Prepare Primer "M" and apply it to the cleaned area as called for in MPD-38001, paragraphs 4. 4. 5 through 4. 4. 8 and 4. 5, using paint spray gun or brush to suit shop requirements. Maximum dry film thickness need not exceed 0. 0005 inch provided there are no bare spots and the entire area of the S-II unit is primed.

4. 4. 2. 3 Continue priming each area until the entire surface of the S-II unit is primed and protected with plastic sheet. Allow the primer to cure for 4 hours at room temperature of 90° F(±5 °) per MPD-38001, paragraph 4. 5. 4.

NOTE: Primer is not detrimental to strain gages.

4. 4. 3 Equipment Setup

4. 4. 3. 1 On the movable platform install sub-base SF-14862 to adapt tool No. SF-14856 with oscillating three-gun arrangement to the platform.

NOTE: Locate sub-base SF-14862 on the platform so that the impinging stream from the guns will be in the radial direction with respect to the tank surface and the gun nozzle will be approximately 54 inches from it.

4. 4. 3. 2 Clean three 3120 nozzles, three 0. 026 inserts, three 0. 035 orifices, and three 0. 046 orifices, for Binks airless spray guns Model 43P, then assemble the guns.

NOTE: At 2300 psi, the output is 5. 4 lb/min; at 3000 psi, the output is 6 lb/min.

4. 4. 3. 3 Two-Gun Setup

Install two guns approximately 54 inches apart (keep the third gun as spare) in the upper and lower gun locations of oscillator SF-14856 (see Figure 4-9). Connect shop air supply to the oscillating and skate air motors, and by adjusting the air flow control valves verify the gun oscillating action and up and down motion of the skate. The gun swing angle settings and location are (Figure 4-9) for the area between Stations 425 and 326. Set oscillating rate at approximately 50 osc/min.

4. 4. 3. 4 The two-gun setup for cylinder no. 2 and bolting ring area (Stations 326 to Station 283) is the same as above except the settings for this area must be checked out on simulator (see Figure 4-10).

NOTE: Dimensions, angles, etc., on Figures 4-9 and 4-10 are approximate and may be varied as required to achieve best results.

4. 4. 3. 5 Prepare two sets of containers of Nopco BX-250-A spray foam components A and B, and connect them to the heaters of Binks type "C" formulators rigged for Nopco Foam. Locate these materials on the movable platform.

4. 4. 3. 6 Fill the pressure pots and let the two components stabilize at room temperature ($80^{\circ}\text{F} \pm 5^{\circ}$). Verify that the nitrogen pressure of 110(± 10) psi in both pots is maintained during all spray operations.

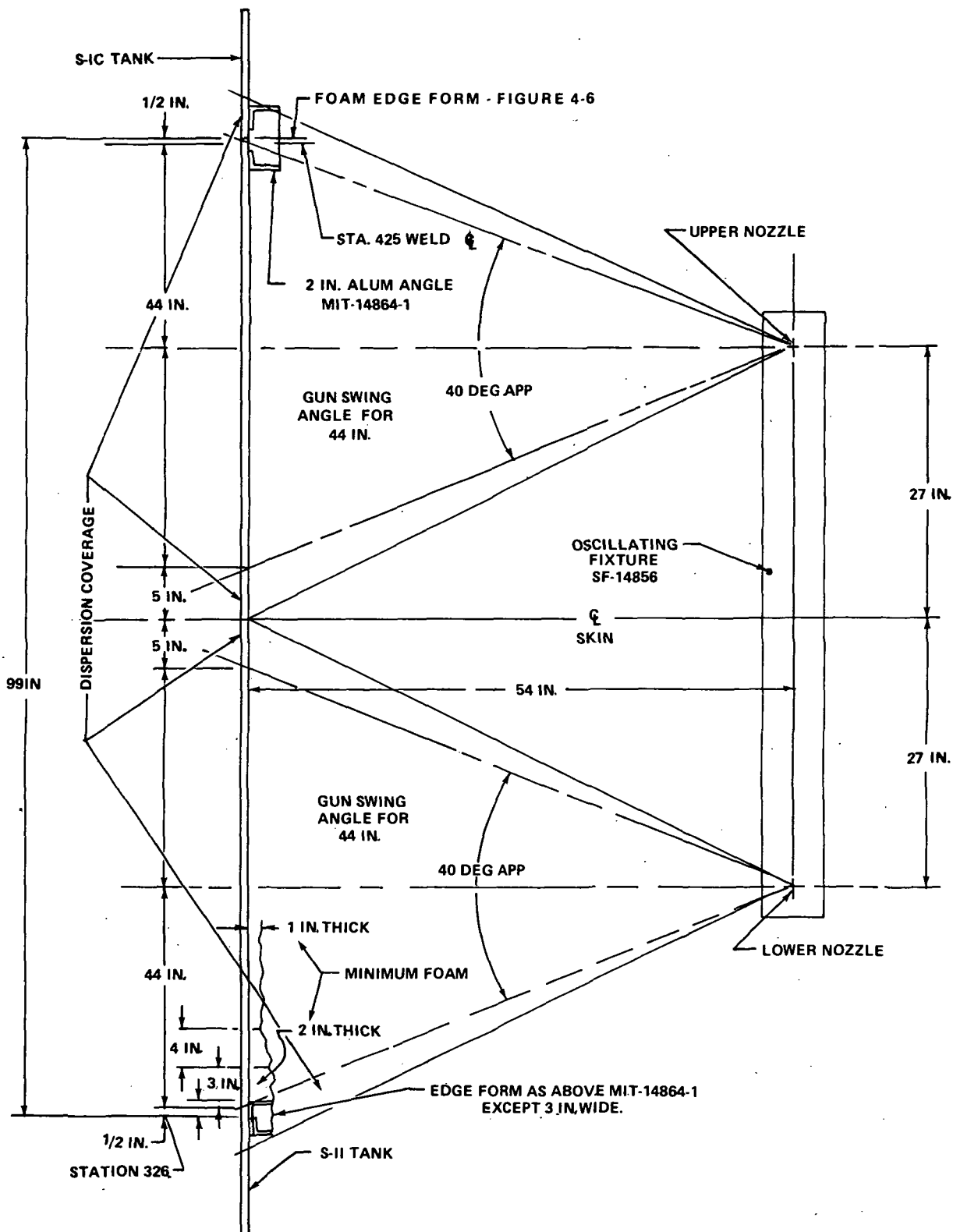


Figure 4-9. S-II sidewall gun seing angle stations 425-32G.

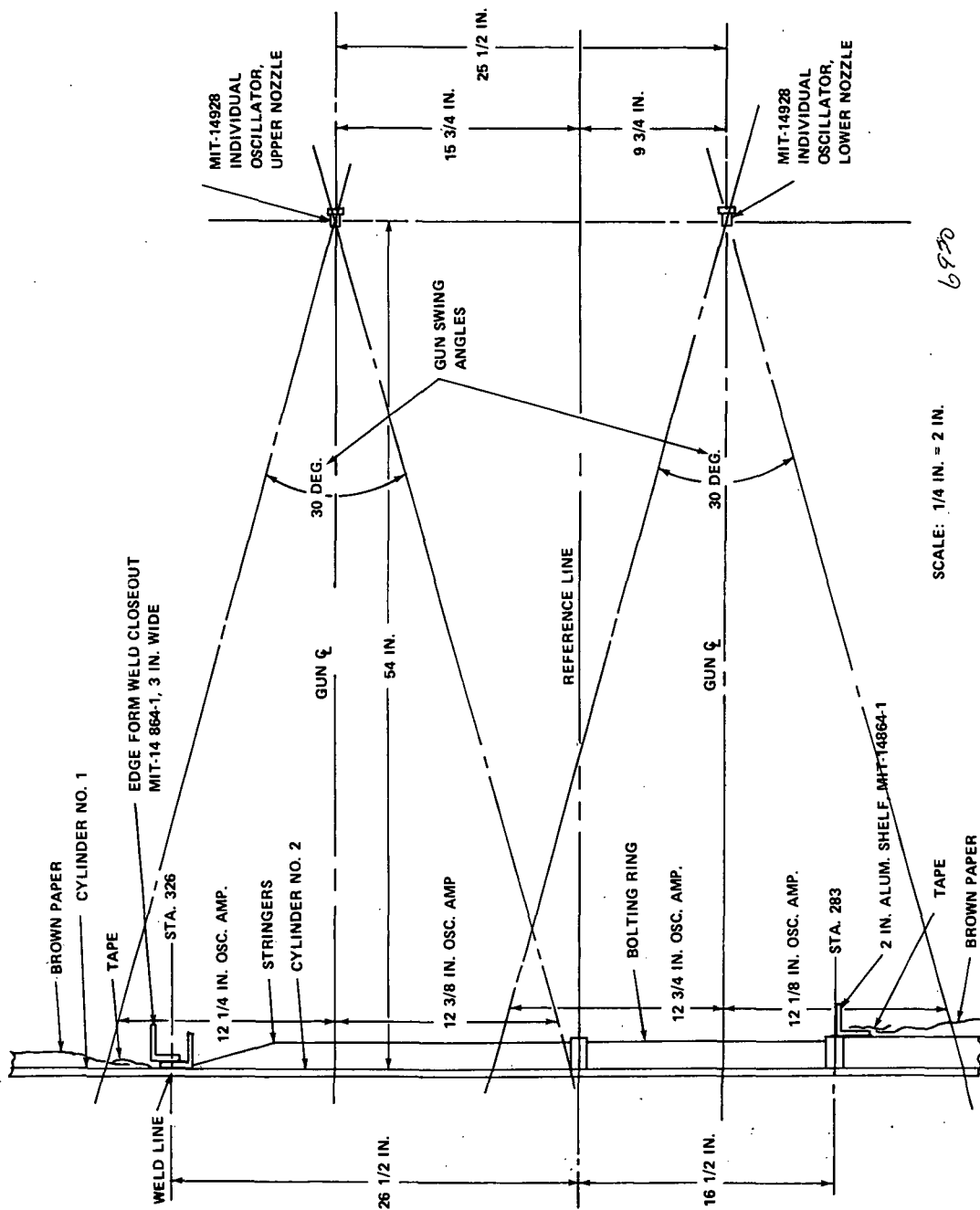


Figure 4-10. Gun swing angles, stations 326-283.

4. 4. 3. 7 Adjust the shop air pressure supply to the air motor driving stirring paddles so that the paddles do not turn in excess of 30 rpm.

4. 4. 3. 8 Connect the component hoses from the formulator rigged for Nopco BX-250-A foam outlets to the guns.

4. 4. 3. 9 Spray Nopco Foam at room temperature ($80^{\circ}\text{F}\pm 5^{\circ}$).

NOTE: General operating characteristics are further detailed in MPD-38001.

4. 4. 3. 10 In case the bung pumps are used for material supply, the air pressure regulators of both pumps should be set at approximately 30 psi, resulting in 110 (± 10) psi on both components. The N_2 blankets are required on drum with a maximum pressure of 3 to 4 psi.

4. 4. 3. 11 Before triggering the guns, prepare three solvent pressure pots by mixing 1 gallon of acetone with the same amount of methanol for each pot and through the solvent (black) hoses connect two pots to the corresponding guns. (Keep the third pot as spare.)

4. 4. 3. 12 Connect the shop air supply to the solvent pot pressure regulator and adjust it to 30 to 40 psi; check that the solvent flows freely through the gun.

4. 4. 3. 13 Purge the gun after each triggering. With guns mounted on the oscillator SF-14856 purging of the guns must be made into a waste bucket.

4. 4. 4 Spray Foaming

4. 4. 4. 1 Two-Gun Setup - Sidewall between Stations 426 and 326 (Weld lines)

A. Set the platform at a convenient height to foam area between these stations.

B. Locate foam edge weld closeouts MIT-14864-1 (Figure 4-6), per Figure 4-9, attach with double-back tape to tank wall to form edge of foam 3 inches below weld at Station 425 and 1 1/2 inches above weld at Station 326. Closeout at Station 326 is 3 inches wide.

C. Tape brown paper to cover area above Station 425 and below Station 326 to prevent overspray adherence.

D. Remove plastic sheeting (used during cleaning) from Sections K-K, R-R, T-T, V-V, Y-Y, and AA-A-A, drawing 30M13991, and install foam barrier tools MIT-14973 in these locations, including sections H-H and M-M below Station 326.

E. Locate foam edge weld closeouts MIT-14864-2 (Figure 4-6) with 30-degree angle on leading side over four vertical welds to form 6-inch wide space. Affix masking tape to the angles to prevent foam adherence inside. See Section G-G and detail "E" of the drawing No. 30M13991.

F. Attach the stop test panels to brown paper or cardboard 120 inches long by 4 feet wide, form hinge with tape and attach to one vertical closeout angle set and fold under (see Figure 4-11), affix start test panels to brown paper or cardboard 120 inches long by 4 feet wide and place over stop area. Place additional start-up paper as required to verify steady gun operation before encountering start test panels.

G. Start equipment (two guns) on paper, spray over the start panels, and proceed around tank without stopping. While spraying, remove start panels and start paper or cardboard, and fold stop panels over on the foam to allow stop panels to be sprayed and guns to stop on paper after completion of spray operation. Turntable rotational speed shall be adjusted to produce a 1-inch minimum foam thickness (approx. 78 in/min, at 5.4 lb/min/gun), which increases to 2 inches at 4 1/2 inches above Station 326.

H. Allow foam to cure 4 hours minimum before removing Station 425 edge-forming tools. Carefully trim to release tape and pull edge forms out. Leave Station 326 edge tools in place. Remove brown paper above Station 425.

4.4.4.2 Two-Gun Setup - Bolting Ring Area (Stations 326 to 283)

A. Set cylinder no. 2 and platform at convenient height to foam the bolt area (Stations 326 to 283).

B. Remove brown paper below Station 326 closeout edge-forming tool.

C. Mask-off with brown paper the area above Station 326 and below Station 283 to prevent overspray adherence.

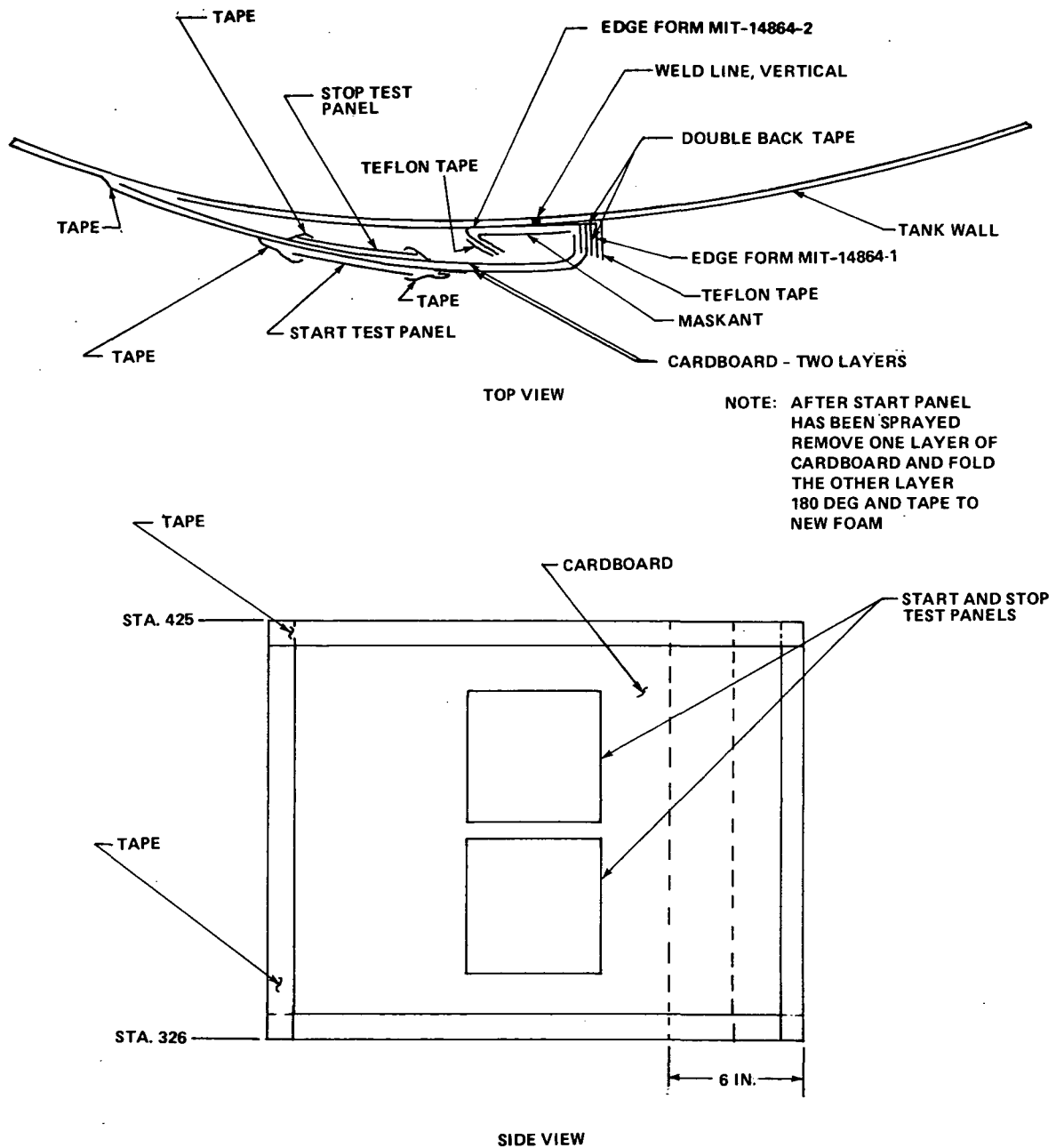


Figure 4-11. S-II Start and stop test panels.

D. Using double-backed adhesive tape and foam edge aluminum angle MIT-14864-1 with Teflon tape, install 2-inch wide shelf at Station 283.

E. Over the four vertical welds between Station 326 to Station 299.38 only, apply Teflon or aluminum-backed tape stopping at outer edge of the adjacent stringers to allow 1/2-inch minimum foam thickness to adhere to the stringer edges. (See Section F-F, drawing No. 30M13991.)

NOTE: These closeouts are 5.4 inches wide - not 6 inches. There will be several sections between stringers where strain gages are installed. These will be covered by Boeing.

F. Position two individual gun oscillators MIT-14928 (quick return constant velocity vertical oscillation unit) 54 inches from surface.

Use in upper gun No. 3140 nozzle, in lower gun No. 2640 nozzle. In both guns use 0.035-inch orifice for component A and 0.046-inch orifice for component B and 0.026-inch sapphire insert. Set gun swing angles to cover 43 inches at 60 oscillations per minute (see Figure 4-12).

NOTE: Dimensions, angles, etc., are approximate and may be varied as required to achieve best results.

G. Affix start and stop panels and spray as called out in 4.4.4.1 paragraphs F and G. Paper or cardboard need only be 60 inches long rather than 120 inches.

NOTE: Spray gun operating pressure should be approximately 2300 psi. Surface speed of the tank must be determined using a simulator provided by R-ME-TP in order to give a minimum of 1/2 inch coverage over the stringers and 2 inches minimum over balance of the area.

H. Allow 4 hours cure time before trimming and removal of edge forms.

4.4.5 Poured and Spray Foam Repairs

4.4.5.1 Repairs and In-Process Rework - Larger than 6-inch diameter

A. Trim out discrepant area leaving a radius at corners of 1-inch or larger. Maintain 90 degree angle between cut foam and tank wall. Clean out foam to tank wall.

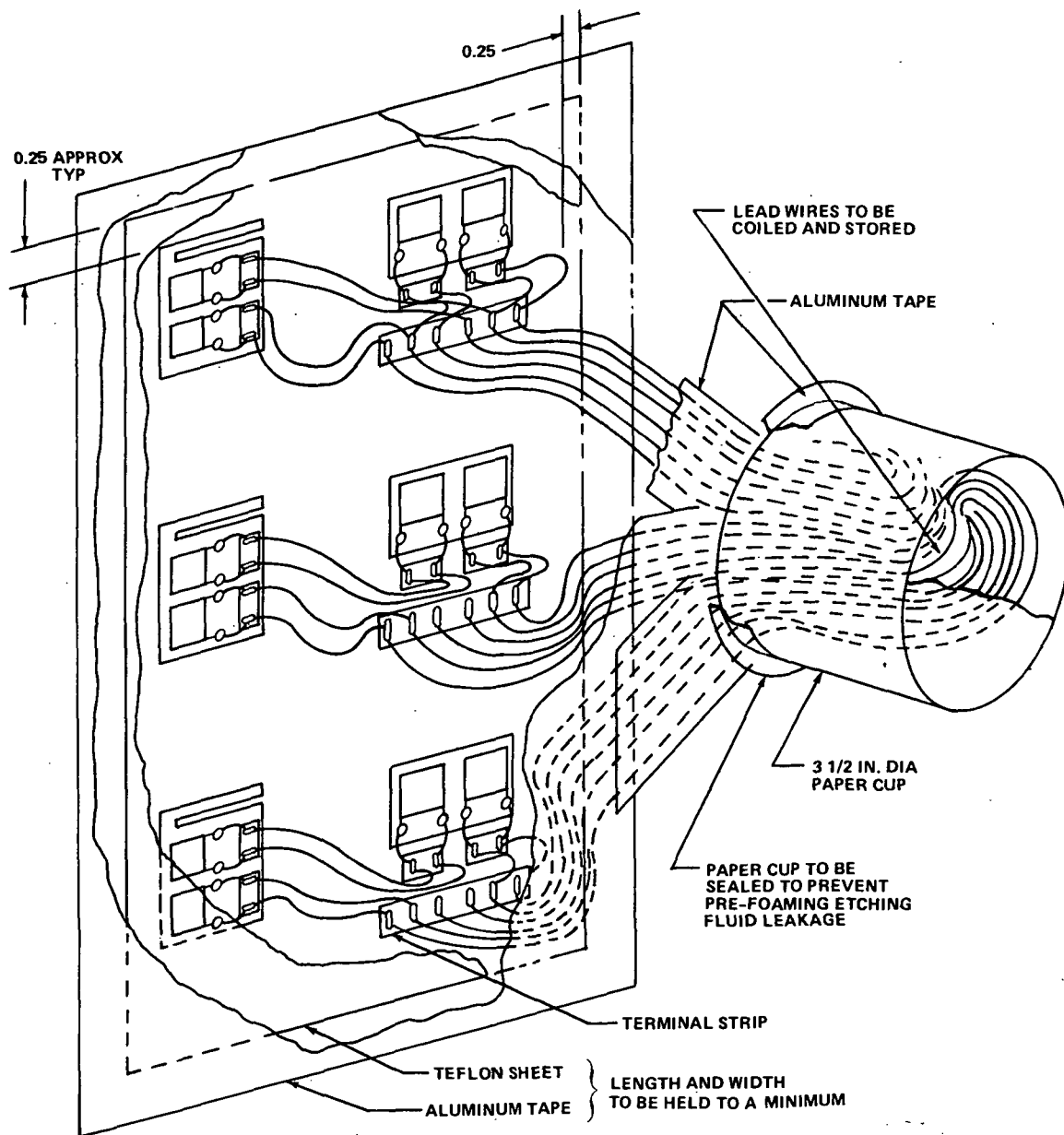


Figure 4-12. Typical masking for 3 and 4 gage clusters.

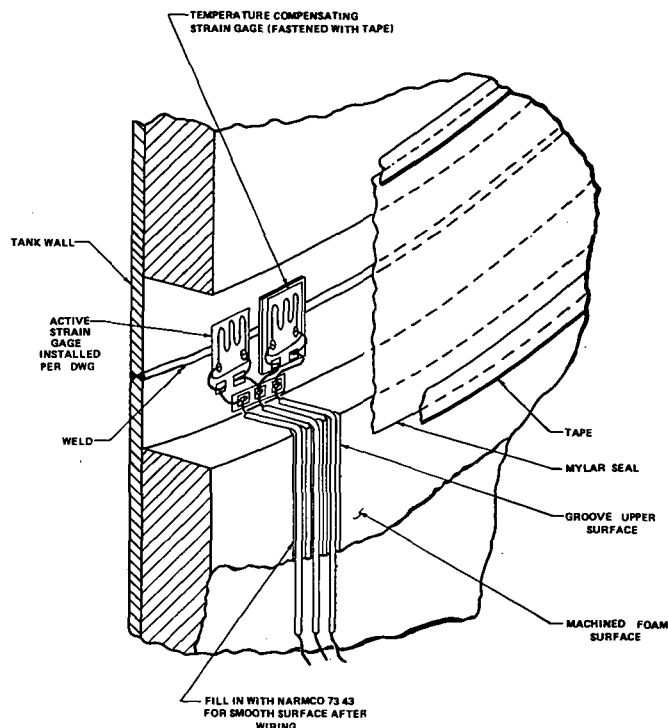


Figure 4-13. Temperature compensating strain gage.

B. Mask edges of cutout with 2-inch wide aluminum tape and brown paper attached to the aluminum tape.

C. Vacuum loose material, lightly sand surface of tank, wipe tank surface with MEK and dry with clean gauze, and apply brush coat of primer "M" (for S-II sidewall) or Z6020 (for S-IC portion). Allow to cure 2 hours minimum.

D. Prepare Adiprene L-100 or Narmco 7343 with Moca curing agent (12.5 percent). (See paragraph 4.2.5.16.) Apply to cut edge of foam, allow to cure 12 hours minimum, 24 maximum.

NOTE: Polyester Resin may be substituted for Adiprene. See paragraphs 4.2.5.16, 4.2.5.17, and 4.2.5.18.

E. Spray foam to fill the open area using vertical oscillation, mechanical or hand. Allow to cure 4 hours minimum, trim to contour of surrounding area.

4.4.6 Strain Gage Treatment-Foamed Area

A. Cover strain gage area with aluminum tape. Protect leads under a paper cup.

B. Seal cups to metal surface to protect the leads from the etching acid (see p. 136).

C. Spray foam over protective cup. After 4-hr cure, carefully trim out cups (see sketch on p. 136).

D. Where use of cups is impractical, pot wires with Narmco 7343/7139 or Adiprene L-100 Moca.

E. Spray or pour foam per 4.4.5.1 or .2, depending on area to be covered.

4.4.6.1 Strain Gage Wire-Weld Areas

A. Place strain gage wire leads under edge forms during spray foaming.

B. After removing edgeforms, press wires into foam to permit routing of wires under Mylar tape (see sketch on p. 137).

4.4.7 Outer Coating

A. Prepare foamed surface for outer coating by machining per specifications.

B. Apply the outer coating per 4.2.7.1 and .2. Allow 48-hr cure.

4.4.8 Close Out Foam Filler Blocks

A. Fabricate foam weld close out filler blocks as required.

B. Coat them with Narmco 7343/7139 or Adiprene L-100 Moca compound per 4.2.7.1.a.

4.4.9 Mylar Covers

A. Apply cleaned 5-mil Mylar Type 500A covers in accordance with specifications.

B. Seal each cover with 2-inch aluminum-backed adhesive tape.

MANUFACTURING PROCESS FOR
CLEANING OF CRYOGENIC INSULATION TEST CONTAINER
AND CONTAINER COMPONENTS

1. SCOPE

1.1 Scope - This manufacturing procedure covers the approved interim Process Engineering Laboratory requirements for cleaning of the cryogenic insulation test container and test container components.

1.2 Applicability - The requirements set forth in this process are applicable to the cleaning of all cryogenic insulation test containers and components.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents of the issue in effect on the date of use of this process form a part of this process.

SPECIFICATIONS

Federal

BB-N-411a	Nitrogen
UU-T-81d (1)	Tags, Shipping and Stock
PPP-T-60 (2)	Tape; Pressure Sensitive Adhesive, Waterproof for Packaging and Sealing
O-T-634a	Trichloroethylene, Technical

Military

MIL-A-00148b (ORD)	Aluminum Foil, Dry Annealed
MIL-A-6091A (ASG)	Alcohol; Ethyl, Specially Denatured, Aircraft
MIL-D-16791C	Detergents, Non-ionic

George C. Marshall Space Flight Center

MSFC-SPEC-164

Cleanliness of Components for Use in
Oxygen, Fuel, and Pneumatic Systems,
Specification for

3. REQUIREMENTS

3.1 General - Not applicable.

3.1.1 When the cryogenic insulation test container must be entered for any reason after it has been cleaned, all personnel entering shall wear clean, lint-free nylon or equivalent clothing, gloves, head, and shoe covers, (as supplied by the Safety Engineering and Supply Company, 617 N. 10th Street, Birmingham, Alabama, Catalogue Number SO #646, or equal) to ensure against recontamination of the container.

3.1.2 All materials, parts, tools, and inspection instruments or equipment taken into the container after the container has been fuel cleaned shall be correspondingly fuel cleaned prior to being taken into a container for use. Equipment shall also be accurately accounted for as workmen enter the container and again when they complete their work and come out of the container.

3.1.3 The cryogenic insulation test container and components, after cleaning by the procedures specified herein, shall meet the cleanliness requirements of MSFC-SPEC-164.

3.1.4 It shall be mandatory to air-purge cleaned test containers with air meeting the requirements outlined in paragraph 3.2.4 when personnel enter to work or open the tank for any other purpose.

3.2 Cleaning and Protective Materials

3.2.1 Deionized Water - The minimum specific resistance of the deionized water shall be 50 000 ohms. The water shall contain no particles greater than 175 microns in any dimension.

3.2.2 Aluminum Foil - The aluminum foil used in the protection of the cleaned insulation test container and components shall conform to specification MIL-A-00148B (ORD) and shall be of the dry annealed type.

3.2.3 Tape - The tape used to secure and reinforce protective covers on the cleaned cryogenic insulation test container and components shall conform to specification PPP-T-60 (2).

3.2.4 Drying or Preservation Gases - Pressurized gases used in drying or preserving the cleaned test container and components shall contain no particles larger than 100 microns in any dimension, the oil content of the gases shall not be greater than 3 parts per million by weight, and the moisture content of the gases shall not be greater than 24 parts per million by volume.

3.2.4.1 Air - Pressurized air used in drying or preservation processes shall meet the requirements of paragraph 3.2.4 above.

3.2.4.2 Nitrogen - Nitrogen gas for use in drying or preservation processes shall conform to Type I, Class I, Grade B of Specification BB-N-411a, except that the particle size, oil content, and moisture content requirements of paragraph 3.2.4 above shall be met.

3.2.5 Non-ionic Detergent - The non-ionic detergent used in the cleaning of the cryogenic insulation test container and components shall conform to the requirements for Type I of Specification MIL-D-16791C.

3.2.5.1 Dowell F-33 is the only non-ionic detergent that has been approved for use at the present time. (It is obtained from Dow Chemical Company, Midland, Michigan.)

3.2.6 Trichloroethylene for Flushing or Immersion Degreasing - The trichloroethylene used for flushing or immersion degreasing shall meet all of the requirements for Type I of Specification O-T-634a, except that the non-volatile residue content shall not be greater than 0.010 gram per 500 milliliters when tested as specified in paragraph 4.4.2.2 of MSFC-SPEC-164, and maximum particle size shall be no greater than 175 microns.

3.3 Component Cleaning

3.3.1 Aluminum Alloy Components (Fill and Drain Line) Cleaning

3.3.1.1 Wipe exterior of tubing assembly until completely free of visible contamination using ethyl alcohol and clean, lint-free paper towels or chamois skin.

3.3.1.2 Rinse alcohol from exterior of tubing assembly with deionized water.

- 3.3.1.3 Dry the exterior of the tubing assembly with pressurized air or nitrogen.
- 3.3.1.4 Fill tubing assembly one-half full of clean Type I trichloroethylene at room temperature. Cap the open end of the tubing. Care should be exercised to avoid contamination of exterior surface.
- 3.3.1.5 Place the tubing assembly in a horizontal position and rotate slowly and carefully for 15 to 20 minutes.
- 3.3.1.6 Empty trichloroethylene from tubing assembly.
- 3.3.1.7 Repeat paragraphs 3.3.1.4, 3.3.1.5, and 3.3.1.6 above. Reclean the exterior surface if it has been contaminated by the preceding operations.
- 3.3.1.8 Dry the interior of the tubing assembly by blowing 180°F ($\pm 20^\circ$) air or nitrogen into the open end of the tubing until free of liquid and fumes.
- 3.3.1.9 After drying, the tube shall be protected by sealing all the tube openings and covering the entire surface with a minimum of two layers of aluminum foil secured with tape. The entire component should then be heat sealed in a polyethylene bag that has been purged with the gas used to dry the component.
- 3.3.1.10 Other aluminum alloy components such as the manifold, etc., scheduled to be processed in the existing facility shall be cleaned as specified in Manufacturing Specification MS 100.9, "Cleaning of Miscellaneous Components." After cleaning, they shall be packaged as specified in paragraph 3.3.3.5 below.

3.3.3 Stainless Steel Components (Discrete Level, Temperature, and Pressure Probes) Cleaning

- 3.3.3.1 Immerse temperature probes and all other stainless steel components in an ultrasonic bath containing 0.1 percent non-ionic detergent (Dowell F-33, or equal) solution at 175°F ($\pm 5^\circ$) for 5 to 10 minutes.
- 3.3.3.2 Rinse all components by immersing in deionized water at 75°F ($\pm 5^\circ$) for 5 to 10 minutes.
- 3.3.3.3 Immerse all components in Type I trichloroethylene at room temperature for 15 minutes.

3.3.3.4 Dry each probe by blowing with 180° F ($\pm 20^\circ$) air or nitrogen until free of all liquid and fumes.

3.3.3.5 After drying, temperature probes and all other stainless steel components shall be protected by sealing all openings with a minimum of two layers of aluminum foil secured with tape, placing the sealed probes or components in polyethylene bags, purging the bags with the same gas used in drying the components, heat sealing the polyethylene bags, and packing the bags as necessary to prevent recontamination or damage to the probes during storage.

3.4 Container Cleaning

3.4.1 Preparation for Internal Cleaning (Horizontal Position, Prior to Hydrostatic Tests)

3.4.1.1 Prior to starting internal cleaning operations, confirm the following:

A. Safety Requirements - Before any personnel enter the container, there shall be assurance of a safe, adequate supply of air flowing into the container.

B. All parts or components that require internal installation on the container shall be completed before internal cleaning.

3.4.1.2 Full compliance with paragraphs 3.4.1.1 and 3.4.1.1B above is mandatory since no fabricating operation of any kind shall be performed inside the container after final cleaning and drying, unless specifically authorized by an engineering document. In the event such approval is granted, the entire container shall be recleaned by the method outlined in this specification, upon completion of the additional internal fabricating operation.

3.4.2 Internal Container Precleaning (Horizontal Position, Prior to Hydrostatic Tests)

3.4.2.1 Remove all grease or wax pencil marks by wiping with ethyl alcohol using clean paper towels or chamois skin. Air dry (allow to evaporate to dryness in room air).

3.4.2.2 Lightly tap, with a rubber mallet, in the area adjacent to all spot welds (within 1 inch) on the external surface of the container, until the entire circumference has been tapped.

3.4.2.3 Inside the container directly opposite the point where tapping has been done on the outside, use a 0.002-inch feeler gage between the container skin and the spot-welded leg of the zeering stiffener to locate and remove any foreign material, dirt, and/or expulsion particles from the spot-weld operation that may be lodged between the contacting container wall and stiffener surface.

3.4.2.4 Vacuum clean the interior of the test container to ensure complete removal of all large-particle contamination.

3.4.3 Internal Container Cleaning (Container in Vertical Position, with Manhole Opening in Bottom Position) after Hydrostatic Tests

3.4.3.1 Attach the Turco nozzle (or equal) to the high pressure stainless steel pipe. Extend the nozzle and pipe up through the manhole and position the nozzle in the center of the container. Attach the hat fixture to the manhole, permitting a positive pressure of approximately 5 psi to be maintained in the container at all times with air preheated to the temperature of the solutions entering the container ($145 \pm 10^\circ\text{F}$). The air used for pressurizing the container shall meet the requirements of paragraph 3.2.4, except there shall not be any moisture content requirements.

3.4.3.2 Spray a 0.1 percent detergent and tap-water solution (Dowell F-33 or equal) into the container at 200 (± 20) psi, at a minimum flow rate of 35 gallons per minute, and at 145°F ($\pm 10^\circ$).

3.4.3.3 Spray clean with the nozzle in the center, fixed position for a minimum of 1 hour and 35 minutes.

3.4.3.4 After completion of detergent spray cleaning and with the container remaining in the vertical position, open deionized water valves. Spray rinse the container interior for a minimum of 1 hour with a spray of deionized water at 145°F ($\pm 10^\circ$), flowing at a minimum rate of 35 gallons per minute and at a pressure of 200 (± 20) psi.

3.4.3.5 After 1 hour of deionized water spray, but with water spray continuing, tap the area adjacent to each spot weld (within 1 inch) with a rubber mallet. Continue the deionized water spray for 30 minutes after tapping, thus providing a total of 90 minutes for rinse time.

3.4.3.6 From a continuous flow of deionized water through the sampling line (a funnel-shaped flange and tube located at the bottom of the container and incorporating a tap and throttle valve) take a minimum of five 500 ml samples. The samples shall be taken at evenly spaced intervals during the entire rinsing cycle. Examine samples and record results.

3.4.3.7 Sample fluids during the various stages of the cleaning procedure and determine whether or not the samples meet or fail to meet cleanliness requirements for the container or components involved.

3.4.3.8 In order for the container to be accepted, five consecutive samples taken in paragraph 3.4.3.6 shall meet the following requirements:

- No particles greater than 2500 microns in any dimension.
- One particle between 700 and 2500 microns for each square foot of surface area to be exposed to the fuel medium.

3.4.3.9 As soon as all desired test samples have been taken, purge inlet line, take inlet line air sample, and remove bottom bulkhead flanges. Inner surfaces of the test container and protruding tubing shall be thoroughly dried by passing 175°F ($\pm 10^\circ$) air through the container and tubing until free of all liquid medium.

3.4.3.10 If the particle count is in excess of the requirements set forth above, repeat the high pressure deionized water spray as specified in paragraph 3.4.3.4, until the particle count meets all requirements of paragraph 3.4.3.8.

3.4.3.11 Upon receipt of information from the Quality Division that all particle size requirements have been met, proceed promptly with paragraph 3.4.3.12.

3.4.3.12 Remove all special flanges and gaskets from bulkhead opening and immediately seal all openings with a minimum of two thicknesses of aluminum foil secured by tape.

4. ACCEPTABILITY PROVISIONS

4.1 Particle Size Determination (Deionized Water)

4.1.1 Particle size and quantity determination shall be in accordance with MSFC-SPEC-164, paragraphs 4.4.1.4.1.(a), (b), and (c).

4.1.1.1 Deionized water shall contain no particles larger than 175 microns in any dimension and when sampled at the sampling point nearest the spray nozzle shall have a pH within the range of 6.0 to 8.0.

4.1.1.2 A 500 ml sample shall be taken at the nearest practicable point to the spray nozzle. Specific resistance of the deionized water, sampled at the sampling point nearest the spray nozzle, shall not be less than 50 000 ohms.

4.1.1.3 Requirements of paragraphs 4.1.1.1 and 4.1.1.2 above shall be met by the general supply of deionized water before pumping equipment is connected, or if previously connected, before valves are opened into the header assembly that supplies the spray nozzle.

5. PREPARATION FOR DELIVERY

5.1 Preservation and Packaging - The test container shall have all openings sealed with approved LOX-clean covers secured in such a manner as to prevent detachment or damage during handling, storage, or shipment. The container shall be protected during shipment in such a manner that absolutely no mechanical damage to the container is likely to occur. The cover shall be designed to facilitate container venting and to prevent recontamination of the container interior.

5.2 Marking - Cleaned fuel containers shall be marked with tags conforming to Type C, Specification UU-T-81, which shall contain the following information:

- Container or identification number
- Contractor identification
- Method of cleaning and micron level
- Date of cleaning
- Title, date, and number of this specification
- Service medium or intended use of container
- Manufacturer's serial number

6. NOTES

6.1 Intended Use - This manufacturing specification, developed by the Process Engineering Laboratory of the George C. Marshall Space Flight Center, is intended for use in the cleaning of the cryogenic insulation test container and container components.

6.2 Any deviation from this basic procedure will require written concurrence from the Process Engineering Laboratory. An emergency concurrence may be made by telephone, but it must be confirmed in writing.

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING SPECIFICATION FOR
CLEANING OF ALUMINUM AND PLASTICS FOR
USE IN ADHESIVELY BONDED STRUCTURES

1. SCOPE

1.1 Scope - This manufacturing specification covers the approved PE Laboratory requirements for cleaning of aluminum and plastics prior to adhesive bonding of honeycomb sandwich structures for use in space launch vehicle applications.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this specification, form a part of this specification to the extent indicated herein.

SPECIFICATIONS

Federal

O-S-595A (3)	Sodium Dichromate, Technical Grade (Sodium Bichromate) Sodium Dichromate Dihydrate
O-S-809A	Sulphuric Acid, Technical
O-T-634A	Trichloroethylene, Technical
QQ-A-255A (1)	Aluminum Alloy Plates and Sheets, Alclad 2014 (R301, Clad 14S)
QQ-A-283A	Aluminum Alloy, Plate and Sheet, 7075
QQ-A-287A (1)	Aluminum Alloy, Plate and Sheet, Alclad 7075

QQ-A-335C (1)	Aluminum Alloy, Plate and Sheet, 2024
QQ-A-362B (1)	Aluminum Alloy, Plate and Sheet, Alclad 2024
ZZ-G-421A (5)	Gloves, Rubber, Surgeons
<u>Military</u>	
MIL-C-7438C	Core Material Aluminum, for Sand- wich Construction
MIL-P-8013	Plastic Materials, Glass Fabric Base, Low Pressure Laminated, Aircraft Structural
MIL-C-8073A	Core Material, Plastic Honeycomb, Laminated Glass Fabric Base, for Aircraft Structural Applications
MIL-A-9067C	Adhesive Bonding, Process and Inspection Requirements for
MIL-A-9180A (1)	Aluminum Alloy, Plate and Sheet, 7178
MIL-A-9183B (1)	Aluminum Alloy, Plate and Sheet, Clad 7178 (ASG)
MIL-P-17667A (1)	Paper, Wrapping, Chemically Neutral
MIL-A-19070A	Aluminum Alloy, Plates and Sheets, 5086 (K 186)
MIL-A-19842B (1)	Aluminum Alloy, Plates and Sheets, 5456

George C. Marshall Space Flight Center

MS 100.2A	Manufacturing Specification for the Deoxidation of Aluminum Alloys
ABMA-PD-A-296	Aluminum Alloy, Plate and Sheet (For Missile Use)
MS 150.0	Manufacturing Specification for Vapor Degreasing of Metallic Surfaces
10419962	CCl_4 Scrubber Method for Analysis of Oil Contamination in Compressed Gases
MSFC-SPEC-164	Cleanliness of Components for use in Liquid Oxygen, Fuel, and Pneumatic Systems

2.2 Other Publications - The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

American Society for Testing and Materials

D1125-59T	Electrical Conductivity of Industrial Water and Industrial Waste Water (see paragraph 6.2)
D1178-60	Chloroform-Extractable Matter in Industrial Water and Industrial Waste Water (see paragraph 6.2)
D1340-60	Oily Matter in Industrial Waste Water (see paragraph 6.2)

(Copies of the publications listed above may be obtained from the American Society for Testing and Materials, 1916 Race St., Philadelphia 3, Pa.)

Gardner Laboratory, Incorporated

GROM -61	Gardner Glossmeter and Reflectometer Operation and Maintenance Manual
Drawing GS-022-A	Assembly P-35, 85° Sheenmeter Head
Drawing GS-024-A	Assembly, P-15, 45° -0° Reflecto- meter

(Copies of the publication and drawings listed above may be obtained from Gardner Laboratory, Inc., 5521 Landy Lane, Bethesda 14, Maryland.)

3. REQUIREMENTS

3.1 General - No deviation from the requirements of this manufacturing specification shall be permitted without prior written approval of the MSFC initiating activity.

3.2 Materials

3.2.1 Demineralized water - Demineralized water shall be oil free, shall have a minimum specific resistance of 50 000 ohms, shall contain no particles larger than 175 microns in any dimension, and shall have a pH between 6.0 and 8.0.

3.2.2 Sulphuric acid - Sulphuric acid used in cleaning shall be Type I, Class 1 of Specification O-S-809A.

3.3 Cleaning and Surface Treatment Procedures

3.3.1 Clad aluminum facing sheets and structural details

3.3.1.1 Degrease - Vapor degrease in the manner outlined in paragraphs 3.3.1 through 3.3.7 of Specification MS 150.0.

3.3.1.2 Alkaline clean - Alkaline clean and rinse in the manner outlined in paragraphs 3.4.4 through 3.4.6 of Specification MS 100.2A.

3.3.1.3 Cleanliness check - After thoroughly rinsing, check for surface cleanliness by observing for water breaks on the metal surfaces as the rinse water drains from the metal. If the water breaks are observed, repeat paragraphs 3.3.1.1 through 3.3.1.3 until water breaks no longer occur.

3.3.1.4 Etching of metal surfaces - Within 30 minutes after alkaline cleaning, immerse for 5 to 10 minutes at 155°F ($\pm 5^\circ$) in an aqueous sulphuric acid-sodium dichromate solution containing 55 (± 5) percent by weight of demineralized water, 40 (± 5) percent by weight sulphuric acid (1.84 specific gravity), add 5 (± 1) percent by weight sodium dichromate.

3.3.1.5 Rinse - Rinse immediately by spraying with or by immersion in demineralized water at a temperature not to exceed 100°F and having a pH between 6.0 and 8.0.

3.3.1.6 Cleanliness recheck - Upon removal from the rinse step (paragraph 3.3.1.5), recheck surface cleanliness by observing for water breaks on the metal surfaces as the rinse water drains from the metal. If water breaks are observed, repeat paragraphs 3.3.1.1 through 3.3.1.6 until water breaks no longer occur.

3.3.1.7 Dry - Dry the water-break-free metal at a temperature no greater than 150°F by allowing to evaporate to dryness in still air, by blowing with air, in an air oven, or in a vacuum oven until thoroughly dry.

3.3.2 Non-clad aluminum details

3.3.2.1 Vapor degrease - Vapor degrease in the manner outlined in paragraphs 3.3.1 through 3.3.7 of Specification MS 150.0.

3.3.2.2 Alkaline clean - Alkaline clean and rinse in the manner outlined in paragraphs 3.4.4 through 3.4.5 of Specification MS 100.2A.

3.3.2.3 Rinse - Rinse by immersion in room temperature tap water for a minimum of 10 minutes. If water breaks occur after rinsing, repeat paragraphs 3.3.2.1 through 3.3.2.3 until water breaks no longer occur.

3.3.2.4 Anodize - Immediately after the rinse procedure cited in paragraph 3.3.2.3, materials shall be anodized in the applicable manner outlined in Specification MS 150.2A.

NOTE: After anodizing, detail parts shall be rinsed only in water at a temperature of 80°F ($\pm 5^\circ$), shall not be sealed by immersion in hot water, and shall be allowed to air dry at a maximum temperature of 90°F.

3.3.3 Protection of cleaned parts

3.3.3.1 Handling - After cleaning and surface preparation, as outlined in paragraphs 3.3.1 and 3.3.2, parts shall not be touched by bare hands during any operation prior to completion of the adhesive curing cycle. Clean, white, cotton gloves shall be worn during any necessary handling of parts. At the first visible sign of contamination, these gloves shall be removed and replaced by clean gloves.

3.3.3.2 Wrapping - After cleaning and surface preparation, as outlined in paragraphs 3.3.1 and 3.3.2, parts shall be immediately wrapped in clean, neutral Kraft paper to protect them against contamination from any source prior to the beginning of priming or bonding operations. Anodized parts may remain wrapped for a maximum of 24 hours prior to priming or bonding, while parts which have not been anodized must be primed or bonded within 10 hours. If the time limits specified above are exceeded, parts shall be recleaned.

3.3.4. Clad aluminum after partial bonding

3.3.4.1 Oxide removal - The portion or spot to be cleaned shall be manually abraded using aluminum wool or Scotch-Brite aluminum-oxide material until a bright oxide-free surface is obtained.

3.3.4.2 Degrease - Degrease the abraded area by first solvent wiping with methyl-ethyl-ketone; wiping dry with clean, lint-free gauze; then vapor degreasing the part in the manner outlined in Specification MS 150.0.

3.3.5 Bare and anodized non-clad aluminum after partial bonding

3.3.5.1 Oxide removal - Within 8 hours of the scheduled application of the next adhesive, the portion or spot to be cleaned shall be manually abraded using aluminum wool or Scotch-Brite aluminum-oxide material until a bright oxide-free surface is obtained.

3.3.5.2 Solvent wipe - Degrease the abraded area by washing with methyl-ethyl-ketone then wiping to near dryness with clean, lint-free cloths slightly dampened with methyl-ethyl-ketone.

3.3.5.3 Degrease - Vapor degrease the part in the manner outlined in Specification MS 150.0.

NOTE: When the subassembly contains a honeycomb core to which the second or final skin has been bonded, the vent holes shall be temporarily plugged or sealed prior to vapor degreasing.

3.3.6 Unstabilized honeycomb core, as received

3.3.6.1 Degrease - Vapor degrease unstabilized honeycomb core, as received, in the manner outlined in Specification MS 150.0.

NOTE: Observe the following precautions while vapor degreasing:

- The honeycomb core shall be placed in single layers horizontally on a carrier fabricated so as to ensure rapid and thorough draining of condensed trichloroethylene from the core.
- Core shall not be flushed with or immersed in liquid trichloroethylene at anytime.
- Core shall be vapor degreased a second time if visible contamination is apparent after first vapor degreasing cycle.
- Core shall not be used for any structural purpose if insoluble soils are still present after a second vapor degreasing cycle.

3.3.7 Machined honeycomb core

3.3.7.1 Degrease - Machined honeycomb core, either unstabilized or stabilized with a foam filling, shall be vapor degreased in the manner outlined in Specification MS 150.0. The precautions outlined in paragraph 3.3.6.1 shall be observed during the vapor degreasing operation.

3.3.8 Machined Epon 1001 stabilized core

3.3.8.1 Stabilizer removal

3.3.8.2 Vapor degrease - Vapor degrease the core in the manner outlined in Specification MS 150.0.

3.3.8.3 Solvent rinse - Rinse by spraying thoroughly with methyl-ethyl-ketone until all visible traces of foreign matter have been removed.

3.3.8.4 Vapor degrease - Vapor degrease the core for a second time in the manner outlined in Specification MS 150.0. The precautions outlined in paragraph 3.3.6.1 shall be observed during this vapor decreasing operation.

3.3.9 Drying of core after cleaning

3.3.9.1 Positioning - During all drying operations core shall be positioned in such a way that its cells are fully exposed to air circulation so that entrapped or condensed solvent may drain.

3.3.9.2 Foam filled core - Foam filled core shall be dried at a temperature of 225°F ($\pm 10^\circ$) for a minimum of 2 hours.

3.3.9.3 Unfilled core - Bare, unfilled core shall be dried at a temperature of 225°F ($\pm 10^\circ$) for a minimum of 45 minutes.

3.3.10 Reinforced plastic facing sheets and details

3.3.10.1 Degreasing - Degrease the contact surfaces by solvent wiping with clean lint-free cloths saturated with Type I trichloroethylene. Wipe dry with other clean lint-free cloths.

3.3.10.2 Surface roughening - Roughen the smooth glossy contact surfaces of plastics by grit or vapor blasting in the manner outlined in paragraph 3.8 of Specification MS 150.1, or by manual sandpapering with 120- to 180-grit abrasive paper.

3.3.10.3 Dust removal - Remove the dust, created during sanding, from contact surfaces by wiping away with clean lint-free cloths dampened with Type I trichloroethylene or by the use of a suction device. No attempt should be made to clean parts further after sanding dust has been removed.

3.3.11 Reinforced plastic core

3.3.11.1 Degreasing - After cutting to size, the cell wall edges on the faces of the sliced core shall be degreased by wiping over the cut edges with a clean lint-free cloth dampened with Type I trichloroethylene.

3.3.11.2 Dust removal - Any dust, created during cutting, should normally be removed from cell wall edges during degreasing procedure cited in paragraph 3.3.11.1 or may be removed by the use of a vacuum cleaner.

NOTE: The glass fibers protruding from the cut edges of honeycomb cell walls should not be removed along with the dust as they are necessary for forming a good bond when wet laminated to other glass fabric base plastic laminates.

3.4 Cleaning area - The area used for cleaning of aluminum and reinforced plastic face sheets, formed details, and honeycomb core for adhesive bonding shall have an atmosphere which is kept clean by maintenance of the working area cleanliness.

4. QUALITY ASSURANCE PROVISIONS

4.1 General - Due to the extreme importance of the cleaning process in successful bonding, operating personnel shall diligently make all specified process checks at the times and in the manner specified.

4.2 Process checks

4.2.1 Demineralized water - During operation, demineralized water shall be checked daily by operating personnel in the manner outlined in ASTM Standard D1340-60 to assure that it is oil-free. It shall be checked daily in the manner specified in paragraph 4.4.2.1 of Specification MSFC-SPEC-164 to assure that it meets the particle size requirements of paragraph 3.2.1. It shall be checked daily in the manner prescribed in ASTM Standard D1125-59T to determine that its specific resistance meets the required value.

4.2.2 Cleanliness check - Check the cleanliness of facing sheets by one of the two methods specified below.

4.2.2.1 Utilizing a telemicroscope with a crosshair in a rotating eyepiece and with a prism on the objective end, measure the contact angle resulting when a drop of distilled or demineralized water is placed on the surface of a cleaned facing sheet. A low contact angle (10 degrees or less) indicates a surface satisfactorily prepared for adhesive bonding. Any angle greater than 10 deg, 0 min, 0 sec indicates the surface is not satisfactorily prepared for adhesive bonding and must be recleaned by repeating the steps specified in paragraphs 3.3.1 through 3.3.7 until the contact angle decreases to 10 deg or less.

4.2.2.2 Utilizing an accurately-calibrated portable Gardner 45-Degree Reflectometer and an accurately-calibrated portable Gardner 85-Degree Glossmeter, or an approved equal, determine the reflectivity and the gloss of a cleaned aluminum facing sheet immediately after completion of the cleaning procedure outlined in paragraph 3.3. A reflectometer reading of 40 or less, and a glossmeter reading of 70 or more, indicate a surface satisfactorily cleaned for adhesive bonding. Any reflectometer reading higher than 40, and/or a glossmeter reading lower than 70, indicates that the surface is not satisfactorily cleaned for bonding and must be recleaned by repeating the steps specified in paragraphs 3.3.1 through 3.3.7 until the reflectometer reading drops below 40 and the glossmeter reading climbs above 70.

4.3 Cleaning Solution Analyses, pH, and Temperatures - During operation, cleaning solutions used in facing sheet cleaning shall be analyzed at least once each week by operating personnel in order to maintain specified concentrations and pH. The temperatures of cleaning solutions, rinse water, and drying operations shall be determined and recorded during operation to assure that they are maintained within the specified ranges.

5. PREPARATION FOR DELIVERY

Not applicable to this specification.

6. NOTES

6.1 Intended Use - This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use in cleaning of aluminum alloy and reinforced plastic facing sheets and details to be used in bonded honeycomb sandwich structures for space launch vehicle applications.

6.2 Definitions - For the purpose of this specification, only those parts of ASTM Specifications D1125-59T, D1178-60, and D1340-60 (see paragraph 2.2) referring to the actual procedures by which the applicable determinations are made shall apply in the checking of demineralized water or gases.

NOTICE - When government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian:

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity:

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING PROCESS FOR
CLEANING OF S-1C S-II TEST CONTAINER
PRIOR TO APPLICATION OF SPRAY FOAM

1. SCOPE

1.1 This Manufacturing Process Data outlines the procedures to be used in cleaning and removing the oxide from the surface of the test container.

2. EQUIPMENT AND MATERIAL

2.1 Equipment

2.1.1 Scaffolding and ladders

2.1.2 High lift equipment

2.1.3 Paint brushes

2.1.4 Scrub brushes

2.1.5 Push brooms, long handled with stiff bristles or fibers

2.1.6 Compressed air supply

2.1.7 Air-pressure water nozzles for flushing operations

2.1.8 Water hoses

2.1.9 Collection and disposal system for contaminated rinse water

2.1.9.1 Clean wiping cloths

2.2 Material

2.2.1 Water, Deionized

2.2.2 Methyl Ethyl Ketone (MEK)

2.2.3 Phosphoric Acid (85 to 87 percent $H_3 PO_4$)

- 2.2.4 Nitric Acid (70 to 73 percent HNO_3)
- 2.2.5 Hydrofluorosilicic Acid (30 percent H_2SiF_6)
- 2.2.6 Sulfuric Acid (Concentrated) (98 percent H_2SO_4)
- 2.2.7 Sodium Dichromate
- 2.2.8 Cab-O-Sil (Thickening Agent)
- 2.2.9 Masking Tape Platers
- 2.2.10 Plastic sheeting, polyethylene or polyvinyl chloride

3. PROCEDURE

3.1 Preparation of Chemical Cleaning Solutions.

NOTE: The preparation of these solutions demands the exercise of rigid precautions and all contact with the body must be avoided. Exercise all safety precautions such as wearing acid proof safety clothing, hoods, boots, long sleeved rubber gloves, face masks, and work in well ventilated areas or use air supply masks when preparing and applying the acid solutions.

3.1.1 Modified British Etch for Bulkhead and External Walls

NOTE: Prepare this solution in 10- or 12-quart plastic buckets.

3.1.1.1		<u>Volume</u>
3.1.1.1	Add WATER	1476 ml
3.1.1.2	Add NITRIC ACID (70 to 73 percent HNO_3)	378 ml
3.1.1.3	Add PHOSPHORIC ACID (85 to 87 percent H_3PO_4)	1514 ml
3.1.1.4	Add HYDROFLUOROSILICIC ACID (30 percent H_2SiF_6)	226 ml

Yield (Approx) 1 gallon solution

3.1.1.5 Stir until mixed thoroughly.

3.1.1.6 Add CAB-O-SOL* until the desired degree of thickness is obtained. Stir thoroughly.

* NOTE: COB-O-SIL is an inert thickening agent. The more added, the thicker the solution becomes. Various degrees of thickness are desirable on different surfaces. Temperature will cause variations in the amount of agent required to obtain the desired consistency. Thereafter the amount of CAB-O-SIL to add will have to be determined on the site of the application by trial.

3.1.2 Deoxidizer for Bulkhead and External Walls

NOTE: Prepare this solution as needed in 10 to 12 quart plastic buckets.

The following proportions yield 1 gallon of solution.

	<u>Volume</u>
3.1.2.1 Add WATER	2840 ml
3.1.2.2 Add concentrated SULFURIC ACID (98 percent H ₂ SO ₄)	588 ml
3.1.2.3 Add SODIUM DICHROMATE	378 g

Yield (Approx) 1 gallon solution

3.1.2.4 Stir until the ingredients are dissolved completely.

3.1.2.5 Add CAB-O-SIL* until the desired degree of thickness is obtained. Stir thoroughly.

NOTE: * CAB-O-SIL is an inert thickening agent and the more added the thicker the solution becomes. Various degrees of thickness are desirable on different surfaces. Temperature will cause variations in the amounts of agent required to obtain the desired consistency. Therefore the amount of CAB-O-SIL to add will have to be determined on the site of the application by trial.

3.1.3 Deoxidizer for Y-Ring - This solution may be prepared in plastic lined 55-gallon drums.

Add WATER (demineralized) 55 (± 5) percent by weight

Add SULFURIC ACID 40 (± 5) percent by weight
(98 percent H_2SO_4)

Add SODIUM DICHROMATE 5 (± 1) percent by weight

Stir until the solution is complete

3.2 Cleaning of Tanks

3.2.1 After the positioning of the tank, install the effluent collection and disposal systems, and proceed to etch and deoxidize the tanks in three steps.

3.2.1.1 The Bulkhead

3.2.1.2 The Y-Ring

3.2.1.3 The Vertical Cylindrical Wall

3.2.2 Preparation for Etching and Deoxidizing the Bulkhead

3.2.1.1 Plug the weep holes in the Y-Ring with suitable plastic plugs or equivalent.

3.2.1.2 Fill the Y-Ring with deionized water. Place the drip pans in place at top of tank.

3.2.1.3 Tape polyethylene plastic cover in position so that acids and rinse water will drain over the trough formed by the Y-Ring into the drain trough.

3.2.1.4 Carefully hand wipe all external surfaces free of soil and oils using clean wiping cloths moistened with methyl ethyl ketone (MEK) solvent.

NOTE: Be careful to clean to the edges of the strain gauges.

3.2.3 Etching and Deoxidizing of Bulkhead

3.2.3.1 To a convenient predetermined exterior area of the bulkhead, apply the thickened solution of the Modified British Etch with Nylon Bristled Paint Brushes or Plexiglas Spatulas at ambient temperatures for a maximum contact time of 2 minutes.

3.2.3.2 Immediately flush all surfaces thoroughly using large amounts of water until the effluent water has a pH of 6 to 8.

3.2.3.3 To the same area treated above apply a thickened solution of the Deoxidizer for the Bulkhead using Nylon Bristled Paint Brushes or Plexiglas Spatulas at ambient temperatures for a minimum contact time of 5 minutes.

3.2.3.4 Immediately flush all surfaces thoroughly using large amounts of deionized water until the effluent water has a pH of 6 to 8.

3.2.3.5 Proceed to another predetermined area of the bulkhead being careful to prevent overlapping and repeat the cleaning operations outlined in paragraphs 3.2.3 to 3.2.6 until the whole surface of the bulkhead is etched.

Caution: Do not tape over the cleaned areas. It is permissible to tape over primed areas.

3.2.4 Preparation for Etching and Deoxidizing of Y-Ring

3.2.4.1 Remove the plastic sheeting and the holding tape from the Y-Ring area.

3.2.4.2 Carefully hand wipe all the external surfaces and areas not etched previously free of soil and oils, using clean wiping cloths moistened with methyl ethyl ketone (MEK) solvent. Be careful to remove all residual adhesive from the tank.

3.2.4.3 Cover the outside vertical areas of the Y-Ring with polyethylene sheeting to protect them during etching of the inside through areas of the Y-Ring.

3.2.5 Etching and Deoxidizing the Y-Ring

3.2.5.1 Remove the water from the Y-Ring trough. Fill Y-Ring with Deoxidizer.

3.2.5.2 Permit the deoxidizer solution to remain in contact with the inside trough of the Y-Ring for 30 to 40 minutes.

3.2.5.3 Remove the deoxidizer and flush this area copiously with deionized water until the pH of the effluent is 6 to 8.

3.2.5.3 When the Y-Ring trough is satisfactorily cleaned, proceed to etching and deoxidizing the vertical cylindrical walls.

3.2.6 Preparation for Etching or Deoxidizing the Vertical Cylindrical Tank Walls

3.2.6.1 Carefully hand wipe all external surfaces free of soil and oils using clean wiping cloths moistened with methyl ethyl ketone (MEK) solvent.

NOTE: Be careful to clean to the edges of the strain gauges and the weep hole stoppers.

3.2.7 Etching and Deoxidizing the Vertical Cylindrical Tank Walls

3.2.7.1 To a convenient predetermined exterior area of the tank, apply the thickened solution of the Modified British Etch with Nylon Bristled Paint Brushes or Plexiglas Spatulas at ambient temperatures for a maximum contact time of 2 minutes.

NOTE: Be careful to etch and deoxidize to the edges of the strain gauges.

3.2.7.2 Immediately flush all etched surfaces thoroughly using copious amounts of water until the effluent water has a pH of 6 to 8.

3.2.7.3 To the same area treated above apply a thickened solution of the Deoxidizer for the External Walls using Nylon bristled paint brushes or Plexiglas spatulas at ambient temperatures for a minimum contact time of 5 minutes.

3.2.7.4 Immediately flush all deoxidized surfaces thoroughly using copious amounts of deionized water until the effluent water has pH of 6 to 8.

NOTE: Remove the plugs from the weep holes in the Y-Ring as soon as the upper ring is finished.

3.2.7.5 Proceed to another predetermined area of the tank being careful to prevent overlapping and repeat the cleaning operations outlined in paragraphs 3.2.3 to 3.2.6 until the whole surface of the tank is etched.

4. CONTROLS

4.1 Safety

4.1.1 Safety Clothes - All personnel will be provided with, and required to wear, complete acid-proof protective clothing to prevent any contact of the etchant or the oxidizer with the skin. This includes acid impervious coveralls, over-shoes or boots, hoods or head and neck covering, and long gauntlet gloves.

NOTE: All other safety features which apply are included in the safe operating procedures

4.1.2 The operation will be conducted to provide that the Modified British Etch (paragraph 3.1.1) will not be in contact with the metal tank surfaces for a period of time exceeding 2 minutes, and the Deoxidizer (paragraph 3.1.2) will be in contact with the metal tank surfaces for a minimum period of 5 minutes.

4.1.3 Precautions should be taken in preparation of the solutions to make certain that they are prepared properly, in accordance with this procedure.

4.1.4 Polyethylene sheeting and tape can be used because it will withstand the hydrofluorosilicic acid solutions.

PROCESS CONTROL AND OPERATING PROCEDURES FOR
APPLICATION OF NOPCOFOAM BX-250A URETHANE FOAM INSULATION
(REVISION NO. 4)

1. SCOPE

This Manufacturing Process Data covers the Process Engineering Laboratory approved procedures for cleaning, priming, foaming, and sealing cryogenic test tanks. The foam applied by this process will be used for evaluation of its effectiveness as a cryogenic insulation on such vehicles as S-II stage and other tanks. Pertinent to this MPD is MPD "Repairs of Cryogenic Foam Insulation".

2. MATERIALS

- 2.1 Urethane Foam, Component A (T) and B(R) Nopco BX-250A, in drums
- 2.2 Dupont Adiprene L-100 or Narmco 7343 polyurethane elastomer
- 2.3 Moca (4,4' methylene-bis-chloroaniline), or Narmco 7139 curing agent
- 2.4 Methylene dianiline curing agent
- 2.5 DS-1301 adhesive
- 2.6 Acetone
- 2.7 Methanol
- 2.8 Methyl-Ethyl-Ketone
- 2.9 Toluene (Dry)
- 2.10 Ethanol, absolute
- 2.11 Ethyl acetate, urethane grade
- 2.12 Di-methyl-Formamide

- 2.13 Primer Z-6020, Dow-Corning
- 2.14 Primer "M", Furane Plastics, Components B and C (Epocast)
- 2.15 Ortho-Phosphoric Acid, reagent grade
- 2.16 Concentrated nitric acid, reagent grade
- 2.17 Concentrated sulfuric acid, reagent grade
- 2.18 Hydrofluorosilicic acid, reagent grade
- 2.19 Sodium Di-Chromate, reagent grade
- 2.20 De-ionized water
- 2.21 Chem-seal
- 2.22 Cab-O-Sil
- 2.23 No. 116 glass cloth
- 2.24 Ground foam, BX-250A
- 2.25 Polyvinyl chloride or, polyethylene film (5 to 10 mils)
- 2.26 Clean gauze
- 2.27 Wet/Dry sandpaper, 200 grit
- 2.28 Aluminum alloy test panels, approximately $\frac{3}{16} \times 24 \times 24$ inches
- 2.29 Aluminum alloy strip 0.020 inch to 0.032 inch thick and of required length
- 2.30 Brown paper, 36 inches wide
- 2.31 Corrugated paper, 36 to 48 inches wide
- 2.32 2 to 4 inch plater's or waterproof tape
- 2.33 2 to 4 inch masking tape

- 2.34 1/2 to 1 to 2 inch Teflon tape
- 2.35 2 to 3 inch double-back tape
- 2.36 2 inch aluminum tape
- 2.37 Wax free paper cups, 16 ounce size
- 2.38 Upjohn CPR-348 pour foam (if used)
- 2.39 Selectron 5003 Polyester Resin, Pittsburgh Plate Glass Co. or
Hetron 32A, Hooker Chemical Co.
- 2.40 Cobalt Napthanate, Harshaw Chemical Co.
- 2.41 MEK Peroxide, Wallace and Tierman Co.
- 2.42 Lefco-Weld 109 adhesive, Leffingwell Chemical Co.

3. EQUIPMENT

- 3.1 Turntable to suit the vehicle
- 3.2 Handling tools to suit the vehicle
- 3.3 Access stands or scaffolding
- 3.4 Individual gun oscillators, MIT-14928 or equal (SF-14856)
- 3.5 Phenolic fibre or Fiberglas spatulas
- 3.6 Phenolic fibre or Fiberglas knives (MIT-14972)
- 3.7 Nylon bristle paint brushes
- 3.8 Binks Hot Hydraulic Formulator(s) Type "C" modified for 1:1.12
ratio with pressure pot(s) or bung pumps
- 3.9 Binks solvent supply pot(s)
- 3.10 Binks airless spray gun(s), Model 43P
- 3.11 Paint spray gun for primer

- 3.12 Contact pyrometer
- 3.13 Plastic curtains around work area
- 3.14 Ventilating exhaust fan with hose
- 3.15 Environmental temperature and humidity control equipment
- 3.16 Infra-red heating lamps and heat guns
- 3.17 P.Q.V. equipment
- 3.18 Test equipment for determination of foam density, tensile and compressive strength
- 3.19 Breathing air face masks
- 3.20 Protective clothing (acid impervious coveralls, boots, and hoods for head and neck covering)
- 3.21 Gloves (plastic, with gauntlet, and clean cotton)
- 3.22 Plastic buckets, 2 to 3 gallon capacity, PVC
- 3.23 Drain pans to suit the vehicle
- 3.24 Foam barriers, as required, (MIT-14864)
- 3.25 Compressed air (shop supply, 120 to 200 lb/in.²) and hoses
- 3.26 Compressed nitrogen, dry, with pressure regulators and hoses

4. PROCEDURES

4.1 Surface Cleaning

4.1.1 The following procedures shall be used for cleaning all vehicle surfaces to be subsequently spray foamed.

4.1.1.1 Carefully hand wipe the exterior surface free of soil, grease, etc., using a clean gauze moistened with MEK or acetone.

4.1.1.2 Where there are stains or oxides, lightly sand these areas with wet/dry 200-grit sandpaper.

4.1.1.3 Wipe the sanded areas with the same solvent used on the balance of the external surface.

4.2 Surface Preparation

4.2.1 Chemical solutions of the Modified British etching and deoxidizing solutions shall be prepared as outlined below.

CAUTION: Preparation of these solutions demands the exercise of rigid safety precautions and all contact with the body must be avoided. Exercise all standard safety precautions by wearing safety acid-proof clothing, hoods, boots, gauntlet gloves, and face masks. Work shall be performed in a well-ventilated area or use air supply masks when preparing or applying the acid solutions.

4.2.2 The etching solutions shall be prepared in plastic containers by volume using the following proportions:

De-ionized water	1476 ml
Add concentrated Nitric Acid (70 to 78 percent HNO_3)	378 ml
Add Ortho-Phosphoric Acid (85 to 87 percent H_3PO_4)	1514 ml
Add Hydrofluosilicic Acid (30 percent H_2SiF_6)	<u>226 ml</u>
219 in ³ App. 1 gal.	3594 ml

Stir until mixed thoroughly.

4.2.3 Add Cab-O-Sil* and stir until the desired degree of etching paste consistency is obtained.

* NOTE: Cab-O-Sil is an inert thickening agent. The more added, the thicker the solution becomes. Various degrees of thickness are desirable on different surfaces. Temperature changes will cause variations in the amount of agent required to obtain the desired paste consistency. Consequently, the amount of the agent to be added will be determined on the site of the application by trial.

4.2.4 The de-oxidizing solutions shall be prepared in plastic containers by volume to the following proportions:

De-ionized water	2840 ml
Add concentrated sulfuric acid (98 percent H ₂ SO ₄)	588 ml
Add Sodium Dichromate	<u>378 gr</u>
208 in. ³ , App. 1 gal	3428 ml

Stir thoroughly until Sodium Dichromate is completely dissolved and solution mixed.

4.2.5 Add Cab-O-Sil until the desired degree of de-oxidizing paste consistency is obtained.

4.2.6 Secure the drain pan(s) in appropriate location.

4.2.7 Work on a limited area at a time while protecting adjacent area with plastic sheeting held with plater's tape.

4.2.8 Apply the acid etching paste with a nylon brush or a spatula to the cleaned tank surface to a thickness of approximately 1/16 inch. Stir paste frequently while applying. Allow the etchant to react for a maximum of 2 minutes. Then scrape off the paste with a spatula and rinse clean with de-ionized water until the pH value of the effluent is between 6 and 8. Move to a next limited area, being careful to avoid overlapping.

NOTE: If the surface has been treated with Alodine, let the etchant break through Alodine first then start timing for etchant action.

4.2.9 Apply the de-oxidizing paste to the etched area to a thickness of approximately 1/16 inch. Stir paste frequently while applying. Allow the paste to de-oxidize for 5 minutes minimum (45 minutes maximum). Scrape off paste excess with a spatula and rinse thoroughly clean with de-ionized water. Check the pH value of the rinsed surface while wet. The pH level should be between 6 and 8. If it is not, further rinsing shall be done until the pH is of the required value.

4.2.10 Repeat etching and de-oxidizing operations until the entire surface to be foamed is covered.

4.2.11 Air dry all cleaned surfaces. If necessary use a heat gun. Surfaces shall be thoroughly dry for priming operations.

NOTE: Clean cotton gloves shall be worn when handling cleaned or primed parts and machined foam surfaces.

4.2.12 Protect the cleaned and dried surfaces with a plastic sheeting held with masking tape until ready for primer application.

NOTE: The cleaned surfaces shall be primed within 12 hours after completion of cleaning operations.

4.3 Priming

NOTE: The tanks other than S-II stage vehicles will be primed with Z-6020 Dow-Corning primer and S-II stage vehicles will be primed with primer "M" of Furane Plastics.

4.3.1 Priming of Tanks other than S-II Stage Vehicles

4.3.1.1 Prepare a Z-6020 primer by mixing 0.20 gram of primer with 99.8 gram of absolute ethanol. If a larger amount of the primer is required it shall be prepared in the same proportion by weight. Unused primer shall be discarded 8 hours after mixing.

4.3.1.2 Mix the primer solution thoroughly prior to application, and using a paint spray gun apply a thin coat of the mixed primer to all cleaned surfaces to be foamed. Keep shaking the primer container from time to time while spraying.

4.3.1.3 The dried primer film thickness need not exceed 0.0005 inch and shall be cured for a minimum of 1 hour at room temperature (60 to 90 °F).

4.3.1.4 All primed surfaces shall be protected by brown paper or equivalent in preparation for spray foaming.

4.3.1.5 The foam shall be applied to the primed surfaces within 14 days after curing of the primer. If this period is exceeded the surfaces shall be wiped with MEK and reprimed as specified in paragraph 4.3.1.

4.3.2 Priming of the S-II Stage Vehicles

4.3.2.1 Prepare the epocast primer "M" by thoroughly mixing 1 part of component "C" with 80 parts of component "B" by weight. Unused primer shall be discarded 8 hours after mixing.

4.3.2.2 Prepare thinner for the primer by mixing 30 parts of MEK with 70 parts of Toluene by weight.

4.3.2.3 For brushing use 100 parts of resin primer mixed with 100 parts of thinner by weight.

4.3.2.4 For spraying mix 100 parts of resin primer with 180 parts of thinner by weight.

4.3.2.5 Wipe the cleaned surfaces of the vehicle to be foamed with MEK prior to applying primer "M".

4.3.2.6 Brush or spray the wiped surfaces with the prepared primer "M" to a maximum dried film thickness of 0.0005 inch and allow the primer to cure for a minimum of 4 hours at room temperature (60 to 90° F).

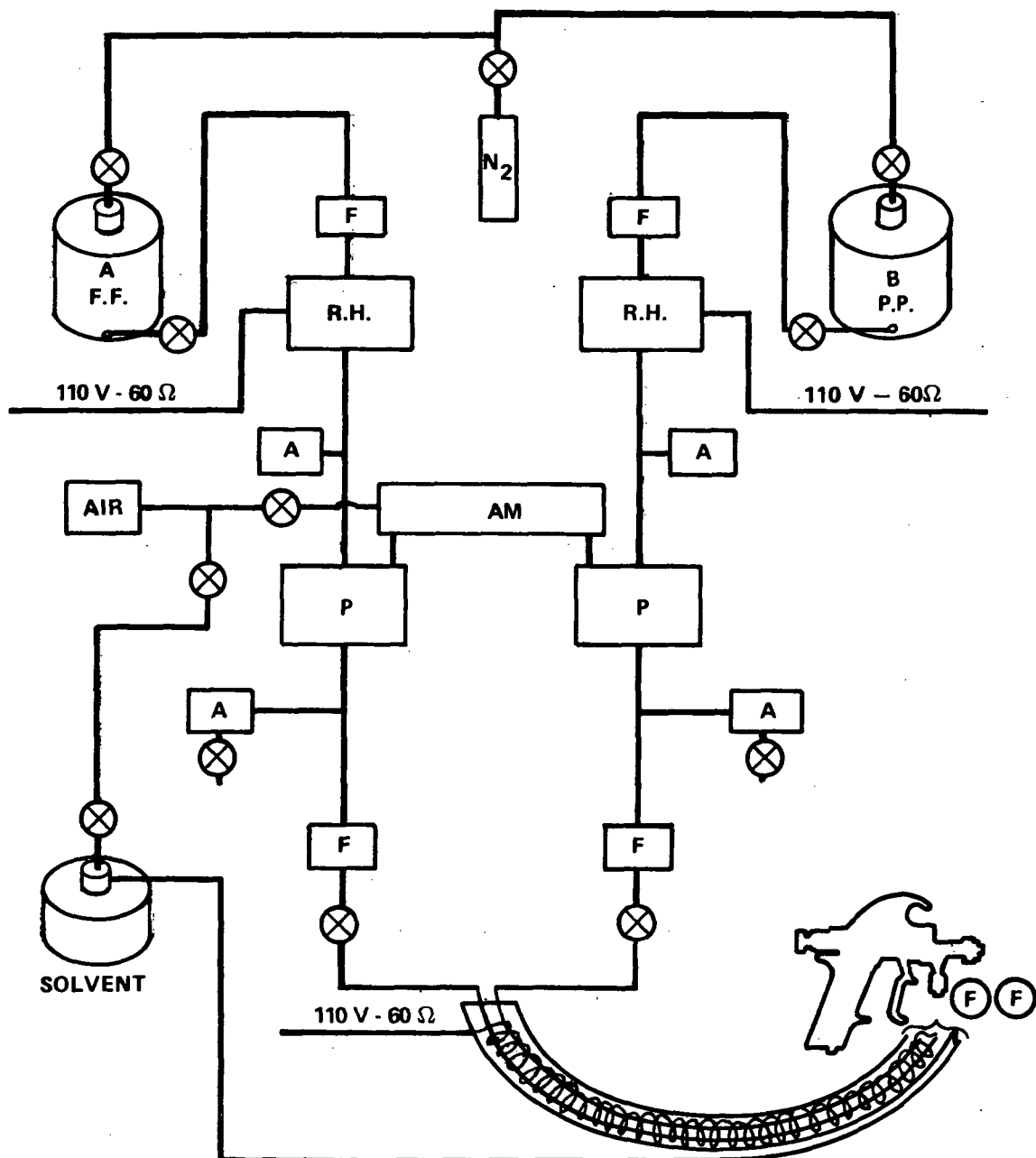
4.3.2.7 All primed surfaces shall be protected with brown paper or plastic sheeting in preparation for spray foaming.

4.3.2.8 The foam shall be applied to the primed surfaces within 14 days after curing of the primer. If this period is exceeded, the primed surfaces shall be wiped with MEK and reprimed as specified in paragraph 4.3.2.

4.4 Equipment Setup

4.4.1 Depending on size and configuration of a vehicle to be spray foamed, one, two, or three individual spray gun arrangement can be used. Accordingly, one, two, or three sets of Binks Type C Hot Hydraulic Formulators, modified to have 1:1.12 output ratio shall be set up for operation either by hand or automatically.

4.4.2 Each Binks formulator set shall include pressure pots or bung pumps (Lark or Falcon), accumulators, filters, solvent pump(s) with appropriate hoses, valves, pressure regulators, tracing tape heater(s), and airless spray gun(s) Binks Model 43P. For the setup see block diagram on Figure 4-14.



P.P. - PRESSURE POT

A - ACCUMULATOR

F - FILTER

R.H. - RESIN HEATER

P - PUMP

AM - AIR MOTOR

Figure 4-14. Hot hydraulic spray foam unit diagram as modified.

NOTE: The support fixture SF-14856 with adapter SF-14862 mounting one, two, or three oscillating guns may be preferred to the individual oscillators MIT-14928 for automatic operation, depending on circumstances.

4.4.3 Ordinarily the gun(s) will be assembled with cleaned nozzle No. 3140, 0.026 inch sapphire insert, and 0.035 inch orifice for component A (T) and 0.046 orifice for component B (R). However, these sizes may be varied as required by conditions and vehicle configuration.

4.4.4 Check general condition of the Binks equipment, paying particular attention to the nuts of the piston pump attachments.

4.4.4.1 Check that solvent pot(s) is filled with 50 percent methanol and 50 percent acetone (1-gallon minimum). Connect the 5-foot hose between Formulator Air Control and air pressure regulator of the solvent pot. Adjust air pressure to 20 (± 5) psi, this will result in 500 to 625 psi of the solvent.

4.4.4.2 Check nitrogen supply in high pressure bottle to assure adequate amount to pressurize the system.

4.4.4.3 Check that the pressure pots are filled with Nopco components A (T) and B (R) and that relief valves are set for approximately 115 psi; connect nitrogen supply hoses to regulators, tighten "C" clamps and adjust nitrogen pressure to 110 (± 10) psi on both pots.

4.4.5 When new 55-gallon drums of components A (T) and B (R) are to be opened, the specific gravity ratio of both components shall be determined prior to use of the materials and at intervals specified by R-QUAL as follows:

4.4.5.1 The material drums shall be rolled, tumbled, or some other method used to thoroughly mix the material.

4.4.5.2 Temperature of both components shall be 70° to 74° F.

4.4.5.3 Determine specific gravity of both components by standard hydrometer method.

4.4.5.4 Specific gravity of component A should be 1.229 to 1.241, specific gravity of component B should be 1.196 to 1.220.

4.4.5.5 In the event that the material is supplied to the formulator directly from drums and bung pumps (Lark or Falcon), the air pressure regulators on the pumps shall be set at approximately 30 psi resulting in 110 (± 10) psi on both components.

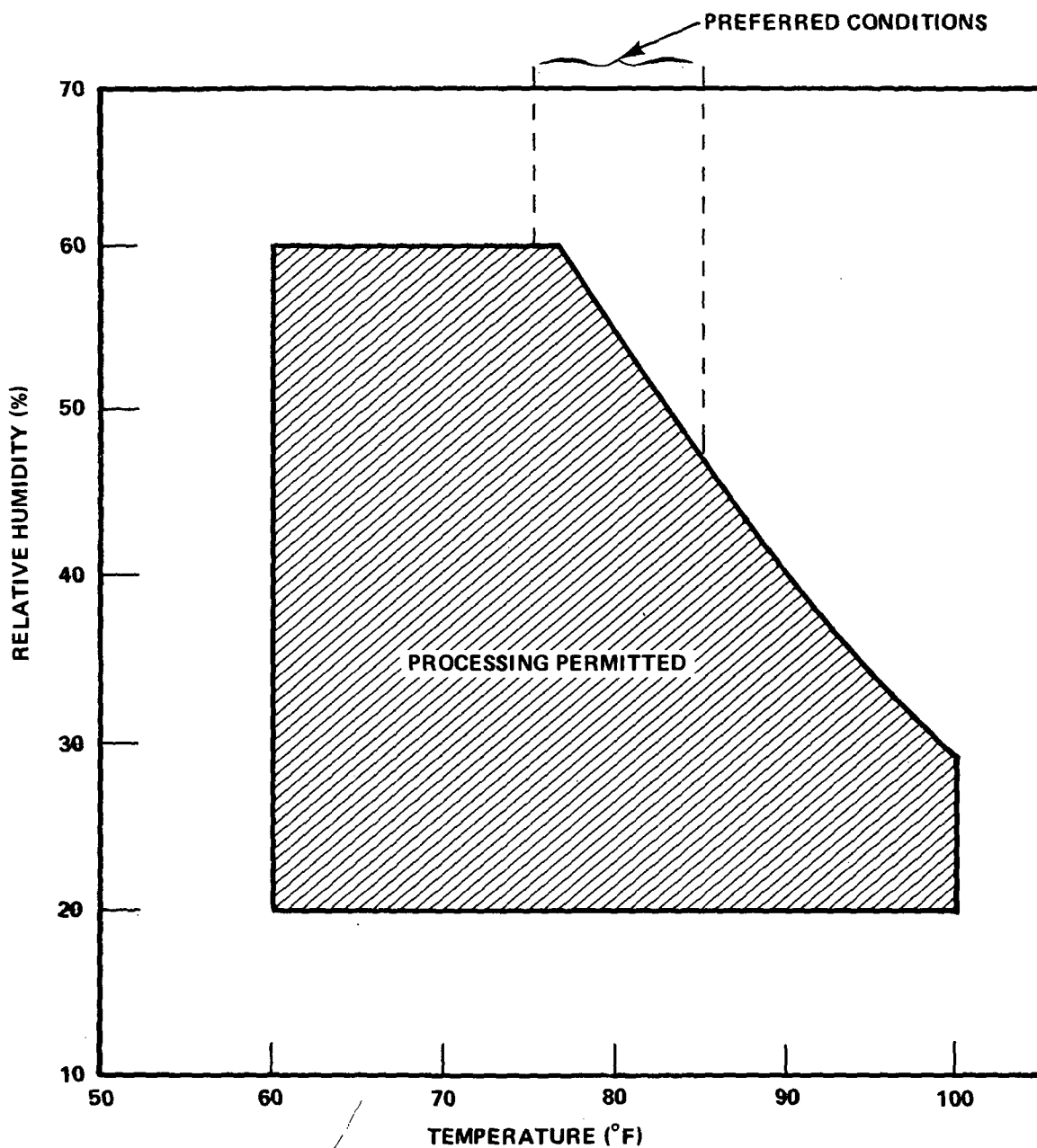


Figure 4-15. Temperature versus relative humidity requirements.

4.4.5.6 Connect shop air supply through the largest available diameter hose (1/2-inch minimum) to the air control valve of the formulator, and from there to the air motors on top of the pressure pots driving stirring paddles. Adjust air pressure for the motors so that the paddles do not rotate at more than 30.

4.4.5.7 Connect the formulator output hoses and the solvent supply hose to the gun, along with the tracing heat tape and its control.

4.4.5.8 Check that solvent flows freely through the gun.

4.5 Spray Foaming Preparation

4.5.1 Spraying of urethane foam shall be performed within a protective enclosure with environmental control and ventilation. All standard safety procedures shall be followed.

4.5.2 Check the relative humidity and temperature recorder for proper conditions. The ideal limits for spray foaming are: 80°F for temperature and 50 percent or less for humidity. See Figure 4-15, Psychrometric chart, for allowable limits and preferred conditions.

4.5.3 To insure that the Binks Type C, modified Hot Hydraulic spray foam unit will operate with a minimum pulsations, the following "Machine Filling Procedure" shall be followed for filling the formulator system with the material:

4.5.3.1 Connect "A" and "B" material sources to the respective input with 3/4-inch diameter hoses.

NOTE: This filling procedure shall be used when beginning spray foaming operation with a pumping unit anytime the system has been depressurized.

4.5.3.2 Close the air valve to the formulator pump.

4.5.3.3 Open the valves between material sources and respective heaters.

4.5.3.4 Open drain valves at the bottom of output accumulators and bleed the system until solid, clear stream of material runs out of the valves.

4.5.3.5 Close valves at the bottom of output accumulators.

4.5.3.6 Remove nozzle, impingement orifices, and plugs from the spray gun.

4.5.3.7 Pull trigger on the gun and hold open until a solid, clear stream of material issues from each side of the gun.

4.5.3.8 Release the trigger and flush the gun.

4.5.3.9 Open drain valves at the bottom of the output accumulators and verify that a solid stream of clear material runs out of the valves, then close the valves.

4.5.3.10 Open the air valve to the formulator pump and set it a lowest operable pressure (approximately 20 [± 5] psi).

4.5.3.11 Check weight output ratio of the components. Shop ratio check shall be made when the equipment and the materials are at a temperature of 70 to 75° F. (If ratio check is made at 80° F ($\pm 5^\circ$) the maximum acceptable ratio is 1.08 due to spring change due to temperature).

NOTE: A simultaneous A and B component weight output ratio check is to insure that $\frac{A}{B}$ ratio of 0.91 (± 0.02) is attained. Radical departures from previously determined ratios, that are not attributable to temperature change, indicate equipment malfunction, such as dirty ball valves.

4.5.4 The weight output check shall be performed as follows:

4.5.4.1 Check that 20 (± 5) psi of air pressure is applied to formulator drive pump and solvent pump; check all fittings, lines, and spray gun for liquid leakage.

4.5.4.3 Remove nozzle assembly and orifices from Binks 43P spray gun. Insure that orifices and parts are clean.

4.5.4.4 Turn A and B component ball valves, outside of filters on formulator "C" (modified), to full open position.

4.5.4.5 Obtain two 16-ounce paper cups and position one cup on a gram weighing balance and tare balance to zero point.

4.5.4.6 Hold the spray gun in one hand over a waste bucket and fully depress trigger mechanism to initiate simultaneous flow of A and B foam components. Allow the components to flow approximately 5 to 15 seconds or until all evidence of air in the lines has been cleared.

4.5.4.7 Hold the two 16-ounce cups in the other hand and move rapidly and simultaneously into the A and B components streams. Hold the cups in position without allowing loss of either material for approximately 10 seconds and then release trigger mechanism to shut off flow. Remove cups from front of gun.

4.5.4.8 Immediately clean the gun by opening solvent flush knob. Run solvent for approximately 5 seconds then secure the flush knob.

4.5.4.9 Weigh individual paper cups containing A and B components and record weights to the nearest 0.1 gram.

4.5.4.10 Determine the ratio of A and B component weights. The ratio shall be in the range of 0.89 to 0.93. Record actual ratio.

NOTE: Figure 4-16 shows results of numerous ratio measurements performed with Upjohn CPR-385-D spray foam components A and B. It is seen that ordinates represent variations in component weight ratios as the results of variations in abscissas represented by changes in formulator pump input pressures and corresponding changes in pump strokes per minute when both components are under equal pressure on the suction side (110 (± 10) psi). It is seen that $\frac{A}{B}$ ratio changes are insignificant: with 25 psi pump input and approximately 73 strokes/min, the ratio is 1.053, with 60 psi pump input and 120 strokes/min, and the same pressure on both components on suction side, the ratio is 1.0550 or a difference of 0.2 of one percent.

4.5.5 Adjust the bi-metal thermostats on top of the heaters to deliver both components at 75 - 80° F and set the heat tracing tape control at 80° F.

4.5.6 During the preheat period occasionally trigger the gun to allow both components to flow past the heater thermometers, otherwise they will not read correctly.

4.5.7 Adjust formulator pump air pressure to 110 (± 10) psi.

4.5.8 Operate the gun to equalize the temperature between heater output and the end of the output hose, by spraying on paper or into a waste bucket for a minimum of 30 seconds before making any test or production spray. This applies to foaming start after new filling of the equipment or a start after the equipment was shut down for 8 hours or more.

4.5.9 Check heaters output temperature during gun operation as adjustment may be necessary to obtain the temperatures specified in paragraph 4.5.5 above.

4.5.10 Protect from foam overspray with brown paper or plastic sheeting the areas of the vehicle that will not or are not at present being spray foamed.

4.5.11 For evaluation of foam density and tensile strength, prepare start and stop test panels from the alloy and condition of material as the vehicle to be foamed. Test panels should be approximately $3/16 \times 24 \times 24$ inches and have surface preparation identical with the vehicle.

NOTE: If multiple guns are employed a minimum of one start and one stop test panels shall be fabricated for each gun.

4.6 Spray Foam Application

4.6.1 Upon initiation of spray foaming at any time, the gun shall be operated for 15 seconds minimum into a waste bucket to completely clear the gun of remaining solvent before proceeding with the approximate density check which shall be obtained at the beginning and the end of each foaming operation.

4.6.1.1 Weight in grams of a wax-free container of 1-quart capacity minimum, and its volume in cubic inches shall be determined.

4.6.1.2 Sufficient foam shall be dispensed into the container to cause the foam to rise above the top of the container. The gun shall be flushed. The foam shall be immediately cut flush with top of the container and the weight of foam determined.

4.6.1.3 Approximate density of foam, in pounds per cubic foot, shall be calculated as follows:

$$\frac{\text{weight of foam in grams}}{\text{container volume in in.}^3} \times 3.81 = \text{Foam density in lb/ft}^3$$

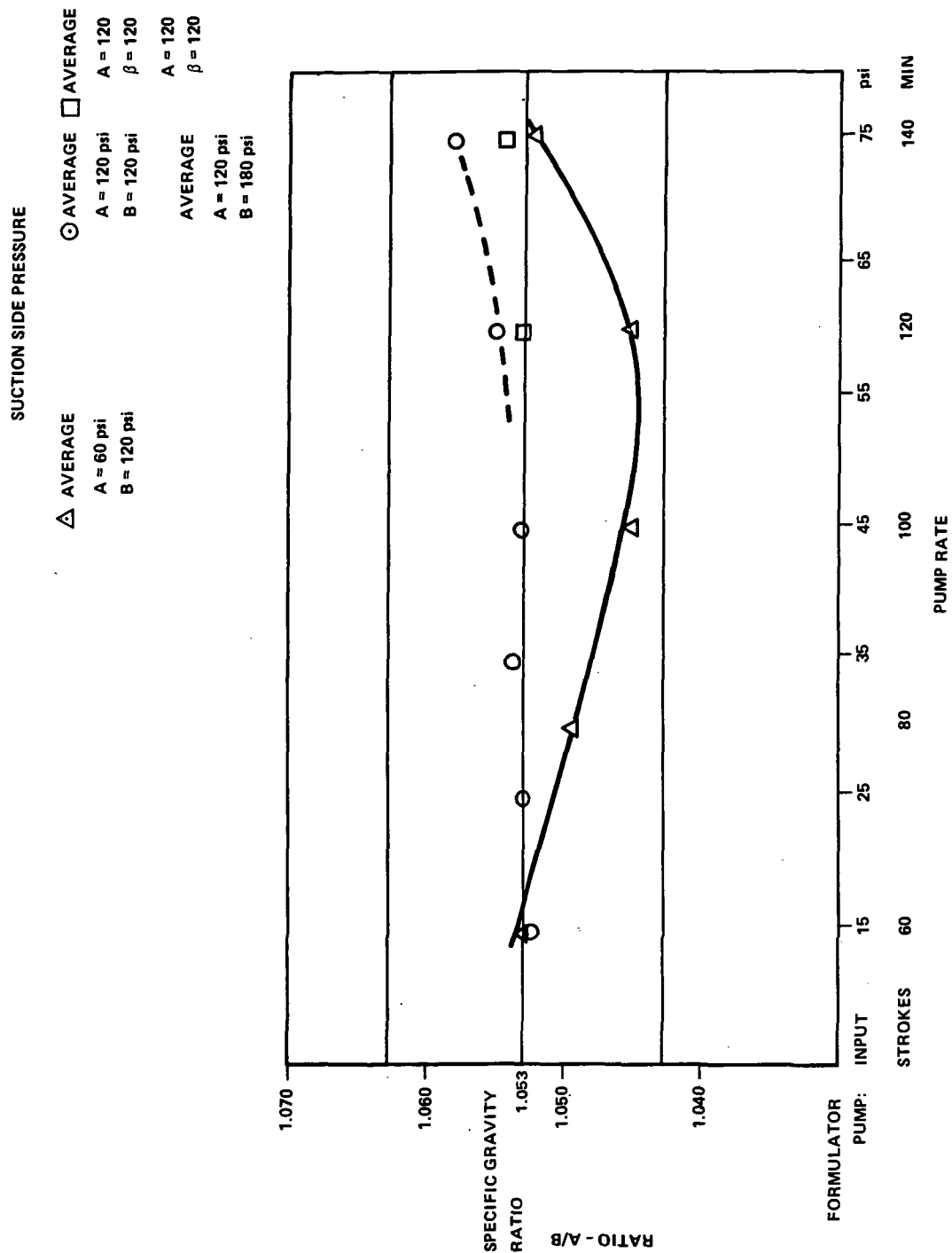


Figure 4-16. Binks formulator — ratio versus pump rate at various supply pressures.

4.6.1.4 If a continuous spray operation is interrupted for a period exceeding 30 minutes, the foam density check shall be repeated.

4.6.2 Secure brown paper and start test panel(s) mounted on corrugated paper just ahead of the beginning of the area to be foamed. Secure stop test panel(s), similarly mounted, with brown paper after the next to the end of the area to be foamed.

4.6.3 With contact pyrometer check the temperature of the surface to be foamed prior to foam application. The temperature should be 75°F ($\pm 10^\circ$). Use heat lamps if necessary.

4.6.4 Start spray foaming onto paper for 15 seconds minimum and transfer the impingement area to the start test panel and then to the vehicle metal without shutdown.

4.6.5 As foaming of the vehicle surface is being completed the impingement area shall be transferred to the stop test panel and terminated on the paper after spraying that panel. Flush the gun after each stoppage.

4.6.6 When additional foaming is required and a delay of 10 minutes or more occurs, the foam interface surface must be coated with polyurethane resin prepared as follows:

4.6.6.1 Melt Narmco 7139 or Dupont Moca curing agent at 250°F ($\pm 10^\circ$).

4.6.6.2 Bring Narmco 7343 or Adiprene L-100 resins to room temperature (65° to 90°F) and mix thoroughly 12.5 parts by weight of curing agent with 100 (± 1) parts of resin. No degassing is necessary. Use 100 percent solids only, do not dilute with a solvent.

NOTE: Selectron 5003 or Hetron 32A polyester resin, prepared with 1/4 percent Cobalt Naphthate and 1/2 percent MDK Peroxide may be used as shop alternate to Adiprene.

4.6.7 Protect vehicle metal surfaces from contact with polyurethane resin by a strip of Teflon tape applied adjacent to foam and extending approximately 1 ($\pm 1/8$) inch onto the foam edges. (This tape will be peeled off after jelling has begun to prevent runs.)

4.6.8 Using a stiff nylon brush apply a uniform coat 0.020 to 0.030 inch thick to all cut or uncut foam surfaces to be foamed.

NOTE: Use softer bristle brush for polyester. Teflon tape is not required. Coverage with polyester is to tank wall.

4.6.9 Allow the resin to cure at atmospheric pressure and 65 to 90° F for 12 to 24 hours. If the 24-hour period is exceeded before additional foaming was begun, another light coat of resin over the first coat shall be applied and allowed to cure for 12 to 24 hours at 65° to 90° F.

NOTE: Polyester cure time is 1 hour minimum after application and 7 days maximum.

4.6.10 The sprayed foam shall be cured at room temperature (65° to 90° F) undisturbed for 4 hours minimum prior to removal of any foam barriers and protective paper.

4.6.11 The foam shall be cured for 16 hours minimum before initiation of any trimming, machining, or sanding operations.

4.6.12 The foam shall be cured for 56 hours prior to application of outer coating or testing of the panels at room temperature for tensile properties.

4.6.13 All test panels are to be tested by R-ME-MW or as directed by R-QUAL which is to maintain records of the fabrication process for permanent reference.

NOTE: The records shall include at least the following: conditions of humidity and temperature, foam test data, results of approximate density checks, mixing ratio, materials operating temperatures, spray equipment type, and number and gun assembly data, also temperature of metallic substrate.

4.6.14 The sprayed foam after curing shall comply with the following requirements:

4.6.14.1 Tensile strength parallel to the rise shall be 35 psi minimum.

4.6.14.2 The apparent density shall be 1.7 to 2.2 pounds per cubic foot.

4.6.14.3 The foam surface shall be free of tack, foreign materials, dents, and gouges.

4.6.14.4 There shall be no cracks in the foam, no separation from attach points, and no evidence of incomplete filling of the foamed areas.

NOTE: If the vehicle being foamed is of circular tank shape and is installed on a turntable, an arrangement for the start and stop test panels similar to the scheme illustrated on Figure 4-17 can be adapted utilizing the following procedure.

4.6.15 Assuming a two-gun operation, mask off with brown paper an area approximately 10 feet high and 8 feet long. (This can be shorter if test panel width is less than 24 inches and height depends on vehicle configuration.)

4.6.15.1 Install with double-backed tape appropriate foam barriers (MIT-14864-1) on top and bottom edges of the exposed area.

4.6.15.2 Checking the gun(s) action for 15 seconds into bucket and then keeping the gun(s) at 54 inches from the surface, start spraying on the paper; spray the exposed area to the required thickness by temporarily slowing down the turntable and terminate spraying on the paper.

4.6.15.3 After 16 hours of foam cure, cut the foam on both vertical edges with a plastic knife, remove the paper (P1), then trim the edges to form approximately 6 inches wide slope (see Step 1, Figure 4-17). Vacuum the edges.

4.6.15.4 Protect metal surfaces from contact with polyurethane adhesive resin by Teflon tape applied at the bottom of the edges as in paragraph 4.6.7.

4.6.15.5 Apply with a stiff nylon brush to the trimmed surfaces the Adiprene L-100/Moca or Narmco 7343/7139 polyurethane adhesive compound prepared as in paragraph 4.6.6.1 and 4.6.6.2. Allow to cure 12 hours minimum, 24 hours maximum.

4.6.15.6 To corrugated paper, tape a strip of aluminum alloy 0,0020 to 0,030 inch thick, approximately 12 inches wide, and extending the entire height of the foamed area. Next to the strip on left side, tape the stop test panel(s) and on the right - the start test panel(s).

4.6.15.7 Tape corrugated paper (P2) with the strip and the test panels over the cured foam from trimmed edge to trimmed edge (see Step 2, Figure 4-17).

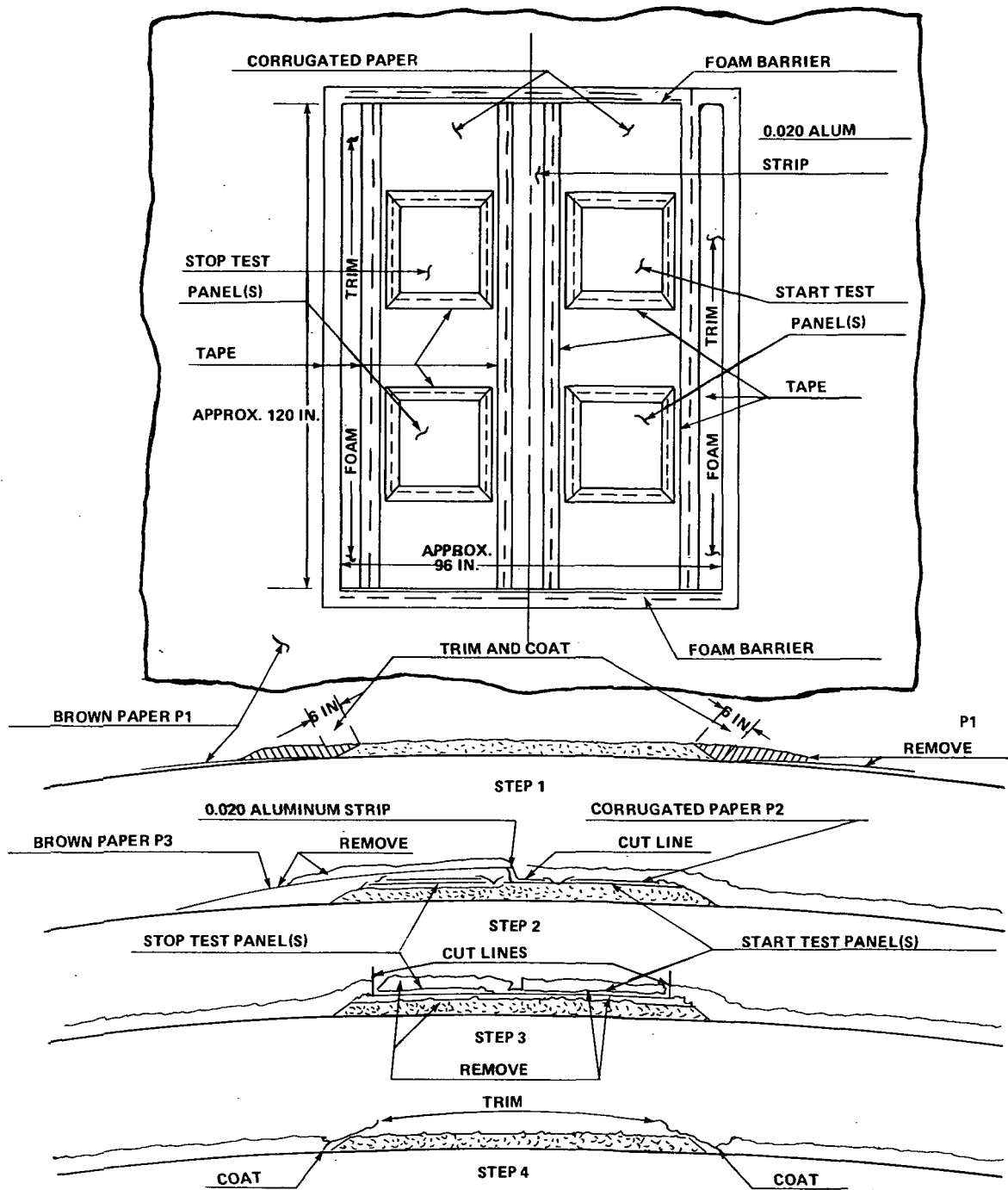


Figure 4-17. Test panel arrangement for a circular vehicle.

4.6.15.8 Tape brown paper (P3), starting with the center strip, over the stop test panel(s) extending the paper beyond the trimmed left edge, to the vehicle metal.

4.6.15.9 After checking the gun(s) action for 15 seconds into a bucket, start spraying over the P3 paper, continue over the center strip, the start test panel, the right trimmed edge, and onto the vehicle primed surface.

4.6.15.10 Prior to completion of spraying the entire circumference of the vehicle, cut the newly sprayed foam with a knife over the center aluminum alloy strip and remove the starting portion of the foam and the P3 paper, exposing the left trimmed foam edge and the stop test panel; spray over both of them and terminate the spraying over the center strips (see Step 3, Figure 4-17).

4.6.15.11 With a plastic knife cut the foam over the left and right edges of corrugated paper P2, being careful to cut to the paper only without penetrating into the originally deposited and cured foam. Remove P2 paper along with the center strip and stop and start test panels. Let cure 16 hours, then cut out the test panels.

4.6.15.12 After 16 hours foam cure, trim off excess of foam over the trimmed edges and remnants of corrugated paper and tapes shown in dotted lines in Step 4, Figure 4-17.

4.6.15.13 Coat newly trimmed surfaces with polyurethane resin adhesive as in paragraphs 4.6.6.1 and 4.6.6.2.

4.6.15.14 Remove foam barriers and coat the newly exposed foam edges also with polyurethane resin compound being careful to first protect the metal surface from contact with the polyurethane resin as in paragraph 4.6.7. Maintain $1 (\pm 1/8)$ inch clearance on all tapered closeouts; all 90-degree closeouts require $1 (\pm 1/16)$ inch clearance.

4.6.15.15 Allow the coated edges to cure 48 hours minimum.

NOTE: For use of polyester resins as shop alternate to polyurethane resins see NOTES in paragraph 4.6.6, 4.6.8, and 4.6.9.

4.7 Spray Equipment Shutdown

4.7.1 When foam application is completed or stopped for lunch or weekend the spray equipment shall be shutdown as follows:

4.7.1.1 Remove gun nozzle nut, nozzle, gasket, insert, and both impinging orifices.

4.7.1.2 Soak and clean these parts in Dimethyl Formamide (DMF) then rinse in MEK or acetone.

4.7.1.3 Clean the gun interior from which the parts were removed by covering front opening with thumb and turning on the solvent flush valve so that the solvent under pressure issues from the orifice openings until mixing chamber and openings are thoroughly clean. Then dry and pack all openings with petroleum jelly.

4.7.1.4 Shut off air supply from main air control valve to the solvent pot pump.

4.7.1.5 Maintain nitrogen blanket over both components in the pressure pots (or drums).

NOTE: Do not release pressure on pots, maintain drum pressure at 2 to 3 psig for safety at all times.

4.7.1.6 Leave all other valves open.

4.8 Foam Trimming

NOTE: Trimming, bonding, and storage of foamed parts without outer coating shall be performed in an area that is maintained at a temperature of 60 to 90°F and relative humidity of 70 percent or less. The uncoated foamed parts being transported from one environmentally controlled area to another shall be sealed in a polyethylene bag, or equivalent, during transportation.

4.8.1 Cured foam surfaces shall be trimmed to the dimension specified on Engineering drawing by cutting, machining, or sanding.

4.8.2 Trimmed, machined, or sanded foam areas shall have a smooth surface free from tears or gouges and the holes shall not exceed 1/16 inch diameter.

4.8.3 Irregularities in the cut foam surfaces shall not exceed 1/4 inch in any direction nor 1/16 inch in depth. Irregularities or discrepancies larger than these shall be reworked or repaired as specified below.

4.8.4 The trimmed foam surfaces shall be vacuum cleaned to remove particles and dust present from trimming operations.

4.8.5 Protect vacuum cleaned surfaces with a plastic sheeting, or equivalent, prior to application of an outer coating.

4.9 Spray and Poured Foam Repairs

NOTE: For repairs and rework utilizing polyester resins see MPD "Repairs of Cryogenic Foam Insulation."

4.9.1 Repairs and In-Process Rework - Larger than 6-Inch Diameter

4.9.1.1 Trim out discrepant area leaving a radius at corners of 1 inch or larger. Maintain 90 degree angle between cut foam and vehicle wall. Clean out foam to vehicle wall.

4.9.1.2 Mask edges of cut-out with 2-inch wide aluminum tape and brown paper attached to aluminum tape.

4.9.1.3 Vacuum clean loose material, then lightly sand surface of the vehicle, wipe it with MEK and dry with clean gauze.

4.9.1.4 For tanks other than S-II stage vehicles prepare Z-6020 primer as in paragraph 4.3.1 and apply a brush coat to the sanded metal surface. Allow to cure for 1 hour minimum at room temperature (60° to 90°F).

4.9.1.5 For the S-II stage vehicles, use primer "M" prepared as in paragraph 4.3.2, applied and cured as in paragraph 4.9.1.4 above.

NOTE: Drain film thickness of either primer should not exceed 0.0005 inch.

4.9.1.6 Prepare Adiprene L-100/Moca or Narmco 7343/7139 polyurethane resin compound as in paragraphs 4.6.6.1 and 4.6.6.2 and with a stiff nylon brush apply a coat 0.020 to 0.030 inch thick to the cut edge of foam. Allow to cure for 12 hours minimum, 24 hours maximum at 65° to 90°F temperature.

4.9.1.7 Before the 24-hour period of curing has expired, spray foam to fill the open area using vertical oscillation, hand or mechanical. Allow foam to cure a minimum of 16 hours prior to trimming to the contour of surrounding area.

4.9.2 Repairs and In-Process - Less Than 6-Inch Diameter

4.9.2.1 Trim out discrepant area as in paragraph 4.9.1.1.

4.9.2.2 Mask, clean, reprime and edge treat as in paragraphs 4.9.1.2 through 4.9.1.6, except cut angle at top to approximately 110 degrees to prevent air entrapment.

4.9.2.3 Mask over the hole to be filled using aluminum tape or thin sheet of aluminum taped in place, leaving a hole at the top for pouring of foam to fill.

4.9.2.4 Pour sufficient foam to fill the discrepant area using Nopco BX-250-A components T (A) and R (B), machine mixed and dispensed directly into the hole to be filled or, using an aluminum template to mask the adjacent foam area, spray the Nopco BX-250-A foam into the hole. If the Upjohn CPR-348 pour foam is available it should be prepared to vendor's instructions and poured from a cup directly into the hole to be filled.

4.9.2.5 Allow the repair foam to cure a minimum of 16 hours before trimming to the contour of surrounding area.

4.10 Protective Outer Coating Application

NOTE: A protective outer coating or sealer shall be applied over the sprayed foam after 56 hours minimum and 7 days maximum of curing after foam application.

4.10.1 Vehicles Other Than S-II Stage

4.10.1.1 Outer coating may consist of two layers of glass cloth (No. 116) impregnated with Adiprene L-100/Moca or Narmco 7343/7139 polyurethane resin adhesive prepared as in paragraphs 4.6.6.1 and 4.6.6.2.

4.10.1.2 Protect metal surfaces with Teflon tape as in paragraphs 4.6.7 from contact with polyurethane resin.

4.10.1.3 Using a stiff nylon brush apply a uniform coat of polyurethane resin compound over the cut and uncut foam insulation, then apply first layer of glass cloth over the compound.

4.10.1.4 Press out with spatula or roll out with a hand roller any bubbles or wrinkles and allow to cure overnight or until the compound jells to prevent runs.

4.10.1.5 Apply another coat of the polyurethane compound and cover with the second layer of glass cloth, again eliminating wrinkles or bubbles.

4.10.1.6 After the compound jells, apply the final coat of the compound over the second layer of cloth.

4.10.1.7 Allow the polyurethane compound to cure for a minimum of 24 hours before moving the vehicle. For a complete cure of the outer coating 7 days are required at room temperature of 65° to 90° F.

4.10.2 S-II Stage Vehicles

4.10.2.1 Outer coating will consist of Chem-Seal material. This material is a two-part system to be prepared per vendor's instructions.

4.10.2.2 Using a paint roller coater apply several coats of this material to the final dry film nominal thickness of 15 mils. One pass dry film thickness is approximately 5 mils.

4.10.2.3 Allow the sealer to air dry at room temperature of 65° to 90° F a minimum of 15 minutes prior to application of each subsequent coat.

4.10.2.4 Prior to application of the final coat of sealer inspect the coated foam surface for voids, those detected shall be filled with a putty prepared as follows:

Mix thoroughly Adiprene L-100/Moca compound with ground BX-250-A foam until workable putty consistency is obtained.

4.10.2.5 Apply the putty mixture to the void areas with a spatula and allow to set for 15 minutes. Scrape off the excess with a spatula or knife.

4.10.2.6 Allow the putty in the voids to air dry for two hours prior to application of the final coat of Chem-Seal.

4.10.2.7 Allow the final coat of sealer to cure at room temperature for a minimum of 24 hours before handling the vehicle.

4.10.3 Outer Coating Without Cloth — When glass (or nylon) cloth is not called for on the Engineering drawing, the polyurethane resin compound can be used alone as an outer coating by brushing or spraying as detailed below:

Method I (paragraph 4.10.3.1) may be used for cut foam areas; Method 2 (paragraph 4.10.3.2) will be used for remaining uncut areas and as additional coating over exposed Method I areas.

4.10.3.1 Adiprene L-100/Moca or Narmco 7343/7139 Cure (Brushing)

A. Prepare Adiprene/Moca or Narmco 7343/7139 polyurethane resin compound as in paragraph 4.6.6.1 and 4.6.6.2.

B. With a stiff nylon brush apply a uniform coat of the resin compound 0.030 to 0.040 inch thick over all cut (machined, sanded, and vacuumed) foam areas and allow to cure for 12 to 24 hours at atmospheric pressure and room temperature of 65° to 90° F.

NOTE: For easier brushing at reduced viscosity, approximately 25 percent by weight of ethyl acetate (Urethane grade) can be mixed at room temperature with the polyurethane resin compound.

4.10.3.2 Adiprene L-100/Methylene Dianiline Cure (MDA, Spraying)

A. Using a cleaned Binks Formulator (1-to-1 ratio) but without heaters, mix by weight in one pressure pot at room temperature 650 parts of Adiprene L-100 and 335 parts of dry toluene.

B. In the other pressure pot dissolve 62.4 parts by weight of Methylene Dianiline (MDA) curing agent in 853 parts of ethyl acetate (urethane grade).

C. Prepare Binks solvent pot by filling it with necessary amount (1 gallon minimum) of ethyl acetate (urethane grade).

D. Prepare a cleaned Binks airless spray gun Model 43P with No. 9-1820 nozzle, 0.020 inch sapphire insert and two 0.035 inch orifices.

E. By-passing the formulator heaters, connect the pressure pots, the formulator, solvent pot, and the gun by cleaned specified hoses. (No heat for tracer hoses is required.) Connect dry nitrogen bottle to pressure pots.

F. Tighten "C" clamps on the lids of pressure pots, check setting of the safety relief valves, and adjust nitrogen pressure on both pots to 110 (± 10) psi.

G. Connect shop air supply (120 psi) to the air control valve at the formulator and adjust air pressure to the air motors driving stirring paddles in the pots so that they do not turn more than 30. Set formulator air pressure at 60 to 100 psi.

H. Adjust air pressure regulator on the solvent pot pump to 30 to 40 psi. Check that the solvent flows freely through the gun.

I. With the vehicle on turntable rotating at suitable velocity, check the gun action into a waste bucket and proceed to spray the outer coating resin compound on all the foamed vehicle surfaces while hand oscillating the gun, holding at 24 to 36 inches from the foam surfaces.

NOTE: Testing the gun action into a bucket is necessary to assure that the solvent has been flushed from the gun.

J. The desired dried film thickness of the outer coating is 10 to 15 mils, while the dry film build-up is about 5 mils per pass, consequently a minimum of two passes are needed, with 25 to 30 minutes drying time between passes.

K. Seven days are required for complete cure of the outer coating of room temperature of 65° to 90° F.

MANUFACTURING SPECIFICATION FOR
ANODIZING ALUMINUM ALLOYS

1. SCOPE

1.1 Scope

This manufacturing specification covers the approved PE Laboratory methods and manufacturing requirements for anodizing aluminum alloy components to be used in space launch vehicles.

1.2 Applicability

The methods and requirements set forth in this specification are applicable to the following aluminum alloys:

1.2.1 Wrought alloys - 1100, 2014, 2017, 2024, 3003, 4032, 5052, 5056, 5086, 5456, 6061, 6063, 6151, 7075, and all clad alloys.

1.2.2 Casting alloys - 13, 13x, 195, 218, 220, 355, 356, 360, and Almag 35.

1.3 Classification

The types of anodizing covered by this specification are:

Type I	- Chromic Acid
Type II	- Sulphuric Acid
Type III	- Hard Coat
Type IV	- Dyed Coatings using Type I or Type II as the basic coating.

2. APPLICABLE DOCUMENTS

2.1 Governmental

The following documents of the issue in effect on the date of use of this specification form a part of this specification.

SPECIFICATIONS

Federal

O-A-51 (C)	Acetone, Technical
O-A-91 (3)	Acid; Oxalic, Technical
O-B-581	Boric Acid, Photographic (H ₃ BO ₃ Crystalline)
O-C-303B	Chromium Trioxide, Technical, Chromic Acid
O-D-306	Diacetone Alcohol, Acetone Free
O-H-795 (2)	Hydrofluoric Acid, Technical
O-O-670	Orthophosphoric, Phosphoric, Acid, Technical
O-S-595 (a)	Sodium Dichromate, Technical Grade
O-S-809A	Sulfuric Acid, Electrolyte, for Storage Batteries
O-T-634A	Trichloroethylene, Technical
BB-C-104 (1)	Carbon-Dioxide, Solid (dry ice)
TT-E-781B	Ethylene Glycol Monoethyl Ether, Technical
TT-I-735	Isopropyl Alcohol

TT-M-268B	Methyl Ethyl Ketone, For use in Organic Coatings
TT-N-97	Naphtha, Petroleum, Aromatic, For use in Organic Coatings
TT-X-916	Xylene

Military

JAN-A-183 (2)	Nitric Acid, 42° Baume'
JAN-T-171 (2)	Toluene
MIL-P-149A	Plastic Coating Compound, Strippable, Hot Dipping
MIL-S-8141 (1)	Strippable Plastic Coating, Sprayable
MIL-L-20209	Lacquer, Strippable, Spray or Dip, For Packaging
MIL-A-8625A	Anodic Coatings, for Aluminum and Aluminum Alloys
MIL-A-13528	Acid, Hydrochloric, Inhibited, Rust-removing

George C. Marshall Space Flight Center

MS 150.0	Manufacturing Specification for Vapor Degreasing of Metallic Surfaces
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STANDARDS

Federal

151A	Metals, Test Methods
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2.2 Other Publications

The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

Turco Products, Inc.

Bulletin No. 84	Smut-Go No. 1 or Turco No. 2897
Bulletin No. 78	Turco No. 4215

(Copies of the publications listed above may be obtained from Turco Products, Inc., P. O. Box 1055, Wilmington, California.)

Wyandotte Chemical Co.

Etchant Bulletin, F-2441	Wyandotte Mil-Etch
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(Copies of this publication may be obtained from Wyandotte Chemical Co., Wyandotte, Michigan.)

Minnesota Mining & Mfg. Co.

Catalogue Sheet	Lead Foil Tape
No. P-ZM-79f-No. 420	

(Copies of this publication may be obtained from Minnesota Mining & Mfg. Co., St. Paul 1, Minnesota.)

Magnaflux Corporation

Bulletin No. 1	Spotcheck Cleaner (Type SKC-2)
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(Copies of this publication may be obtained from Magnaflux Corporation, 7300 W. Lawrence Ave., Chicago 31, Ill.)

Cabot Corporation

Bulletin No. 100

Cab-O-Sil (Silica Aero-Gel M-6)

(Copies of the publication listed above may be obtained from the Cabot Corporation, 77 Franklin St., Boston 10, Massachusetts.)

3. REQUIREMENTS

3.1 General

No deviation from the requirements of this manufacturing specification shall be permitted without prior written approval of the MSFC initiating activity.

3.2 Materials

3.2.1 Acids

3.2.1.1 Chromic acid

This acid shall conform to Specification O-C-303, Type 2, technical grade.

3.2.1.2 Phosphoric acid

This acid shall conform to Specification O-P-313 (1), technical grade.

3.2.1.3 Sulfuric acid

This acid shall conform to Specification O-S-809A, technical grade.

3.2.1.4 Hydrofluoric acid

This acid shall conform to Specification O-H-795 (2), technical grade.

3.2.1.5 Nitric acid

This acid shall conform to Specification JAN-A-183 (2), 42° Baume'.

3. 2. 1. 6 Oxalic acid

This acid shall conform to Specification O-A-91 (3), technical grade.

3. 2. 1. 7 Boric acid

This acid shall conform to Specification O-B-581.

3. 2. 2 Etchants

3. 2. 2. 1 Non-critical

An etchant such as Wyandotte Mil-Etch, or equivalent, at a concentration of 1 ounce per gallon of water shall be used for stripping anodic films from racks and non-critical parts.

3. 2. 2. 2 Critical

A chromic-phosphoric acid stripper shall be provided for stripping anodic films from components. It shall consist of 17 pounds of chromic acid and 3 1/2 gallons of phosphoric acid to a total volume of 100 gallons when added to tap water.

3. 2. 3 Trichloroethylene

The trichloroethylene used shall conform to Specification O-T-634A, Type II.

3. 2. 4 Spotcheck Cleaner

The Spotcheck Cleaner shall be equivalent to that produced by Magna-flux Corporation, Detroit, Michigan.

3. 2. 5 Acetone

Acetone used for all purposes shall conform to Specification O-A-51 (C), technical grade.

3. 2. 6 Pre-stripping solvent

The solvent used for cleaning in paragraph 3. 5. 6 of this specification shall be of the following composition:

Ingredients:	Percent by Volume	
	Minimum	Maximum
Acetone (O-A-51C)	9	10
Toluene (JAN-T-171 (2))	13	14
Isopropyl Alcohol (TT-I-735)	15	16
Xylene (TT-X-916, Grade A)	8	9
Aromatic Petroleum Naphtha (TT-N-97, Type II)	4.0	4.5
Diacetone Alcohol (O-D-306)	5.0	5.5
Ethylene Glycol Monoethyl Ether (TT-E-781B)	5.0	5.5
Demineralized Water	0.0	1.5
Methyl Ethyl Ketone (TT-M-268B)	Balance	

Physical Properties:

Specific Gravity @ 60° F 0.833 - 0.843

Refractive Index @ 25° F 1.4060 - 1.4120

3.2.7 Sodium dichromate

Sodium dichromate shall conform to Specification O-S-595 (A), technical grade.

3.2.8 Demineralized water

Water, possessing a minimum electrical resistivity of 50,000 ohms and filtered to remove particles larger than 175 microns in any dimension, shall be used for anodizing baths.

3.2.9 Maskants

3.2.9.1 Sprayable

This lacquer shall conform to Specification MIL-L-20209.

3.2.9.2 Hot dip

This plastic coating shall conform to Specification MIL-P-149A.

3.2.9.3 Tape

Electroplaters masking tape equivalent to No. 420 lead foil tape made by Minnesota Mining & Mfg. Co. may be used.

3.2.10 Dyes

The following dyes, or their equivalent, are used to produce colored anodic coatings.

- Blue - Aluminum Blue GN, Sandoz Chemical Works, New York, N. Y.
- Green - Naphthol Green, General Dyestuff Corp. , Philadelphia, Pa.
- Red - Aluminum Scarlet C, Sandoz Chemical Works, New York, N. Y.
- Black - Sandoz Black BK, Sandoz Chemical Works, New York, N. Y.

3.2.11 Miscellaneous

A non-silicated, hot alkaline cleaner similar to Turco No. 4215 may be used for degreasing, but it must not etch the alloy or cladding.

3.2.11.1 Nickel acetate or cobalt acetate

The acetates used for sealing colored anodic coatings shall be of reagent quality.

3.2.11.2 Base metal

Metallic parts shall be free from surface defects detrimental to the finish or service of parts.

3.3. Equipment

3.3.1 General

Equipment used in the anodizing of aluminum alloys shall permit the attainment of all quality requirements of this specification.

3.3.2 Cleaning tanks

Tanks of suitable size, constructed of heavy gauge, hot-rolled sheet steel or comparable materials with joints welded inside and outside, shall be used for cleaning parts.

3.3.3 Anodizing tanks

3.3.3.1 Chromic acid process, Type I

Hot rolled, AISI 1020 steel tanks of welded construction, utilizing iron-pipe heating and cooling coils, thermostatic control, and agitation, are suitable for this process.

3.3.3.2 Sulfuric acid processes, Types II & III

Lead-lined, acid-brick lined, or Koroseal lined tanks are suitable for this process. Lead lined tanks may use the lining as a cathode, whereas lead sheets of appropriate area, approximately equal to the areas to be anodized, must be provided for other tanks.

3.3.4 Crane

An overhead, rail-type crane of sufficient capacity shall service the tank area.

3.3.5 Racks

3.3.5.1 Cleaning - Racks for supporting parts during cleaning shall permit thorough draining and may be constructed of steel.

3.3.5.2 Anodizing

Racks, splines, tips or contacts, wire, and perforated containers shall be manufactured of aluminum alloys or titanium.

3.3.6 Agitation

Compressed air distributed by perforated piping is adequate.

3.3.7 Ventilation

An exhaust system shall be provided to eliminate mist and reduce nasal irritations.

3.3.8 Power source

A dc generator or rectifier with rheostat control to permit voltage regulation from 10 to 50 volts and with an indicating voltmeter and ammeter shall be used.

3.4 Solution Preparation

3.4.1 Chromic acid anodizing bath, Type I

- Fill the tank approximately one-half full with demineralized water.
- Add chromic acid flakes to make a 5-percent by weight solution at final volume.
- Agitate until the chromic acid is dissolved.
- Bring solution to final volume with demineralized water and maintain the temperature at 90°F(±5°)

3.4.2 Sulfuric acid anodizing bath, Type II

- Fill the tank approximately one-half full with demineralized water.
- Agitate with compressed air.
- Slowly and cautiously add concentrated sulfuric acid to obtain a 15-percent by weight solution at final volume. With 66° Baume' sulfuric acid, it requires 9.5 gals./100 gals. of water.

CAUTION: Never add water to concentrated acid. Add the acid to the water.

- Increase the solution volume to operating volume and cool to 70°F(±5°).

3.4.3 Sulfuric acid anodizing bath, Type III

This solution is prepared analogously to paragraph 3.4.2 except that a further addition of 2 to 3 ounces of oxalic acid per gallon is made. A commercial hard-coat anodizing addition agent may be substituted for the oxalic acid. The bath can be maintained at 50°F(±5°) when only direct current voltage is used, although temperatures ranging from zero to 20 are common and produce the hardest coats. When ac is superimposed on dc, the bath temperature shall be 20°F(±5°). The temperature reduction is maintained by refrigeration or by the addition of dry ice to the bath.

3.4.4 Dye solutions

Dye solutions shall be prepared in stainless steel or monel tanks using demineralized water. Dyes are to be premixed in a separate container by adding demineralized water at 120° to 140 °F to a weighed quantity of dye. The dye is mixed to a smooth paste and diluted further to make a homogeneous dispersion before adding to the water in the tank. After adding the dye to the tank and stirring, adjust the pH of the dye solution to the range given in Table 4-1 below for the particular dye by using sodium carbonate or glacial acetic acid as necessary.

TABLE 4-1. DYE SOLUTION pH RANGE

Dyes	Concentration	pH
Black (Sandoz)	12 gms./liter	6.0 - 6.5
Blue (Sandoz)	4 gms./liter	5.5 - 6.8
Green (General Dyestuff)	1.0 gm./liter	5.0 - 5.5
Red (Sandoz)	4 gms./liter	5.8 - 6.2
Red (Eaton)	4 gms./liter	4.0 - 5.0

Dye solution temperature shall be maintained at 140 to 160°F.

If other dyes are used, the manufacturer's recommendations shall apply.

3.4.5 Anodic film-sealing solutions

3.4.5.1 Sealing solution for Type I

Hot water meeting requirements of paragraph 3.2.8 at 208 to 212° F, containing 0.01 gram of chromic acid per liter and at a pH of below 7, is adequate for sealing Type I anodic coatings. pH is maintained by additions of chromic acid and/or sodium dichromate.

3.4.5.2 Sealing solution for Type II

A 5-percent by weight sodium dichromate solution in demineralized water at a pH of 5.0 to 6.0 and at a temperature of 208° to 212° F shall be used for sealing.

3.4.5.3 Sealing solution for Type III

A 1-percent by weight sodium dichromate solution in demineralized water at a pH of 5 to 6 and at a temperature of 145° F ($\pm 5^\circ$) shall be used for Type III anodic coatings.

3.4.5.4 Sealing solution for colored anodic films

Color fastness as well as sealing must be effected by operating at the temperature given for each solution in Table 4-2.

TABLE 4-2. APPROVED SEALING METHODS

Composition	pH	Sealing Time (minutes)	Temperature (°F)
0.5% Nickel Acetate	5.5-6.0	5 (minimum)	205 (minimum)
5.6 gm/L Nickel Acetate 1.0 gm/L Cobalt Acetate 8.4 gm/L Boric Acid	5.5-5.8	5 (minimum)	195 (minimum)
5.0 gm/L Nickel Acetate 5.0 gm/L Boric Acid	5.5-6.0	5 (minimum)	195 (minimum)

3.5 Preparation of Parts

3.5.1 General

All metal surfaces to be anodized shall be thoroughly cleaned prior to the application of the anodic coating. Articles made of materials affected by the solutions or interfering electrolytically with the processing shall be masked or not anodized as an assembly.

3.5.2 Tape removal

All tape or remnants of adhesives shall be removed manually and the area cleaned with acetone or other required solvent after removal of the tape from areas to be anodized.

3.5.3 Ink marks

Stenciling inks shall be removed with acetone or other suitable solvent.

3.5.4 Stripping

Any procedure used to remove a previous surface treatment or coating must not damage the base metal.

3.5.4.1 Organic finishes

Stripping of organic finishes such as lacquers, maskants, or paints shall be done with suitable strippers that do not attack the base metal.

3.5.4.2 Anodic films

A. Non-critical tolerance parts - Immerse in a solution of 1 ounce of Turco No. 2897 etchant, or equivalent, per gallon of water at 180 to 200° F.

B. Critical tolerance parts - Immerse in a solution containing 1 pound of chromic acid and 1 pint of 85 percent phosphoric acid per gallon of final solution at 160 to 200° F. It should be immersed for 10 minutes at a time, until the oxide is removed.

C. Rinse - Immerse in flowing cold water until all stripping solution is removed.

3.5.5 Flux removal

Fluxes shall be removed by immersing parts first in hot water for 3 minutes and then by immersing in a solution of 10 percent nitric acid and 0.25 percent hydrofluoric acid by weight at ambient temperature for 3 to 5 minutes.

3.5.5.1 Rinse

Finally rinse the parts to remove all traces of acids.

3.5.6 Preparation prior to welding

When anodic coatings are to be stripped prior to welding, the parts shall be processed as follows.

3.5.6.1 Cleaning

Only the area to be stripped shall be wiped with a clean cloth saturated in the pre-stripping organic solvent given in paragraph 3.2.6 of this specification.

3.5.6.2 Etch

Loosen the anodic coating by applying with a brush a gelatinized LOX-compatible solution of 10 percent nitric acid, 40 percent orthophosphoric acid, 6 percent hydrofluosilicic acid, 5 percent Cab-O-Sil and 39 percent water. Allow the solution to remain 10 minutes. A green smudge will form. Rinse off the etchant with water-dampened cloths.

NOTE: Personnel should avoid contact with this etchant. Rubber or plastic gloves should be worn. If etchant contacts the skin or clothing, wash it off immediately with large quantities of water.

3.5.6.3 Re-etch

Remove the green smudge by brushing the area with a solution of 15 percent sulfuric acid, 10 percent sodium dichromate, and 75 percent water. Repeat, if necessary, to remove all of the green smudge or anodic coating. If smudge still remains, repeat operations in paragraphs 3.5.6.2 and 3.5.6.3.

3.5.6.4 Rinse and dry

Remove the etch with a damp cloth or water rinse, and dry thoroughly.

3.5.6.5 Solvent clean

Immediately prior to welding, clean the area with Spotcheck cleaner or equivalent. If 24 hours have elapsed since completion of this operation, repeat paragraphs 3.5.6.3, 3.5.6.4, and 3.5.6.5 before welding.

3.6 Preparation prior to anodizing

3.6.1 Degreasing

Prior to anodizing, components shall be degreased in accordance with Specification MS 150.0.

3.6.2 Etching

Parts shall be etched in Turco No. 2897 or equivalent when permissible. The concentration of etchant shall be 1 ounce per gallon in water at 180 to 200°F. Etching should continue only until gases are evolved uniformly from the surface. Parts possessing heat treat scale or other dark discolorations will be etched.

3.6.3 Etching-alternate

When etching is objectionable, an inhibited alkaline cleaner at a concentration of 6 ounces per gallon of water at 180 to 200°F for 5 to 10 minutes shall be used.

3.7 Masking

Components requiring preferential anodizing shall be masked to assure complete coverage of areas not to be anodized.

3.7.1 Maskant

Materials used must conform to Specifications MIL-P-149A or MIL-S-8141. Strippable lacquer is to be used where delineation is critical, or where inside surfaces are to be masked. Electroplater's tape is helpful for sharp demarcations. Strippable compound is useful where demarcations are not stringent.

3.7.2 Application

Parts shall be degreased, cleaned, and dried satisfactorily per paragraphs 3.6.1 and 3.6.2 prior to masking. Masking shall be applied to areas not requiring anodizing or only light coatings.

Where practical, areas to be hard anodized should be stopped off with tape, the part immersed in lacquer, the tape removed, and the lacquer touched up with lacquer where necessary.

3.8 Racking

Components shall be racked in such a manner as to insure positive contact under all conditions, to permit circulation of the solution, to prevent air or gases being entrapped in recesses, and to dissipate the heat generated by anodizing.

3.8.1 Contacts

Because of the possibility of burning parts, contacts, tips, or clamps must be clean and possess ample area of contact to avoid high current densities.

3.8.1.1 Precautions

- Avoid clamping or making contacts on highly polished surfaces.
- Aluminum casting alloys containing appreciable silicon may require filing a small contact area thru the casting skin.

- Titanium clamps with anodic coating shall be etched in an aqueous solution of 10 percent by weight hydrofluoric acid. Clamps, other than titanium or aluminum, shall be etched per paragraph 3.5.4.2 B of this specification when required.

3.8.2 Drainage

Components shall be racked to insure good drainage of solutions when removed from the tanks.

3.8.3 Placement of racks in tanks

When racks are positioned on the anode bar, they must clear the sides of the tank, coils, or other cathodic structural elements. It is advisable to place them as near to the tank's center as possible to avoid localized high current density areas.

3.9 Anodizing Procedures

3.9.1 Type I anodic coating

3.9.1.1 Limitation

This process shall not be used on aluminum alloys containing more than 5 percent copper or a total alloy content in excess of 7 1/2 percent, with the exception of 4032 alloy.

3.9.1.2 Loading

Parts which have been degreased and etched, in accordance with paragraphs 3.6.1 and 3.6.2 or 3.6.3 of this specification, shall be loaded on racks, splines, or into perforated containers per paragraphs 3.8 to 3.8.3, and lowered onto the anode bar and into the anodizing bath.

3.9.1.3 Operating bath conditions

The bath composition and temperature shall be as given in paragraph 3.4.1. The voltage shall be slowly applied from 0 to 40 volts over a period of 5 minutes for all alloys except type 4032 aluminum alloy (which contains more than 7 1/2 percent alloy content). In this case the maximum, steady voltage is reduced to 19 volts. After the voltage reaches the maxi-

imum value, it is to be held for a minimum of 30 minutes. The current density shall be a minimum of 1 ampere a square foot to 3 amperes a square foot. The pH of the bath must be maintained between 0.5 and 0.9.

3.9.1.4 Rinse

Rinse the parts in cold, flowing demineralized water.

NOTE: Anodic coatings to be color dyed must not be sealed at this stage and are to be handled per paragraphs 3.9.4.2 to 3.9.4.6 of this specification.

3.9.1.5 Seal

Immerse parts in hot, demineralized water at 180 to 212° F for 5 minutes. The water shall be maintained at a pH of 4 to 6 by addition of chromic acid or sodium dichromate.

3.9.1.6 Dry

The parts may be dried by high pressure filtered air. If oven drying is utilized, dry at maximum temperature of 225° F.

3.9.2 Type II anodic coating

3.9.2.1 Limitations

Type II anodic coatings shall not be applied to welded parts or assemblies which contain joints or recesses from which solutions may not drain. These parts should receive Type I anodizing or a conversion coating. Almag 35 castings shall receive Type II anodic coatings only, followed by immersion in a 5 percent, by weight, oxalic acid solution at room temperature (75°F) for 15 minutes.

3.9.2.2 Loading

Parts which have been degreased and etched, in accordance with paragraphs 3.6.1 and 3.6.2 or 3.6.3 of this specification, shall be loaded on racks, splines, or into perforated containers per paragraphs 3.8 to 3.8.3 of this specification and lowered onto the anode bar and into the anodizing bath.

3.9.2.3 Operating bath conditions

The bath composition and temperature shall be as given in paragraph 3.4.2 of this specification. The voltage shall be adjusted to maintain a current density of approximately 12 amperes per square foot of anode area for 30 minutes and shall be determined per paragraph 4.3.1.2.2 of Specification MIL-A-8625A. Shut off power at the end of the period and remove parts immediately.

3.9.2.4 Rinse

Rinse the parts in cold, flowing, demineralized water.

NOTE: Anodic coatings to be color dyed must not be sealed at this stage and are to be handled per paragraphs 3.9.4.2 to 3.9.4.6 of this specification.

3.9.2.5 Seal

Immerse parts in a hot aqueous solution of 5 percent by weight sodium dichromate. The temperature of the bath is to be 210°F(±2°) and the pH is to be maintained at 5.0 to 6.0.

3.9.2.6 Rinse and dry

Rinse the parts in hot water at 180 to 212°F and blow dry with filtered air or dry in a recirculating oven operating at 210°F(±5°).

3.9.3 Type III anodic coating

3.9.3.1 Limitations

The hard coating produced in this process is more abrasion resistant than Type I or II, but it is more brittle and has less shock resistance. Care should be exercised in its choice and it is limited to aluminum alloys not containing more than 3 percent copper or 7 percent silicon.

3.9.3.2 Loading

Parts shall be prepared and loaded as given in paragraph 3.9.2.2 of this specification.

3.9.3.3 Operating bath conditions

A. dc power - When only dc voltage is applied, a voltage of 10 to 50 volts and a current density of 25 to 40 amperes per square foot are required to produce an anodic coating 0.001 to 0.002 inch thick for an immersion time of 30 (± 10) minutes. The bath composition and temperature shall be as given in paragraph 3.4.2 or 3.4.3.

B. dc power plus ac power - When ac power is superimposed on dc power, a voltage indicator shall be used which registers the forward and reverse voltage. The dc voltage shall be applied within 1 minute up to 15 volts. The ac voltage shall be subsequently applied within 1 minute up to 3 volts. The forward voltage should then be indicated as 9 volts and held for 1 minute before increasing the dc volts to the value in Table 4-3 for the desired coating thickness. The bath composition shall be as given in paragraph 3.4.2 or 3.4.3, and the temperature shall be 20° F ($\pm 5^\circ$). The current density desired is 175 amperes per square foot when area of parts can be measured.

NOTE: (1) Total surface area should be measured accurately to establish the proper amperage. If this is not practical, the process can be controlled by the initial voltage and then by amperage.

NOTE: (2) Hard anodic coatings resist the passage of electrical current, the resistance is proportional to the thickness. As the coating thickness increases, the voltage must be increased to maintain amperage. While final voltage is related to coating thickness, a micrometer must be used to accurately control coating thickness.

NOTE: (3) High current density is desirable to produce the coating thickness in a minimum of time.

NOTE: (4) Vigorous agitation must be employed to prevent local overheating. Localized heating causes the coating to dissolve which will lower the resistance of the area and cause excessive current density to result in this area. Damage can occur in seconds and it is recognized by a rapid rise in amperage. Hard anodizing baths must be closely supervised.

NOTE: (5) Only one alloy at a time may be hard anodized.

TABLE 4-3. TABLE OF HARD ANODIZING VOLTAGES (APPROXIMATE VOLTAGE
TO CONTROL AMPERAGE FOR COATING THICKNESS)

COATING THICKNESS

Alloy	0.003 Inch (Low Density)			0.004 Inch			0.0015 Inch			0.002 Inch		
	Starting Voltage	Time (minutes)	Final Voltage	Starting Voltage	Time (minutes)	Final Voltage	Starting Voltage	Time (minutes)	Final Voltage	Starting Voltage	Time (minutes)	Final Voltage
2014	18	2	19	18	5.5	19	18	8	21	18	11	24
2024	14.5	2.5	15	14.5	8	22.5	14.5	12	33.5	14.5	16	41
6061	14	2	15	14	5.5	18	14	8	21	14	11	26.5
7075	13.5	2	14.5	13.5	5.5	18	13.5	8.5	21	13.5	11	23
356	14	1.5	16	14	4.5	19	14	6.5	20	14	9	21

Alloy	0.003 Inch			0.004 Inch			0.005 Inch		
	Starting Voltage	Time (minutes)	Final Voltage	Starting Voltage	Time (minutes)	Final Voltage	Starting Voltage	Time (minutes)	Final Voltage
2014	18	16.5	30	18	22	35	18	27	40
2024	14.5	23.5	49	14.5	31	56	14.5	39	65
6061	14	16.5	35	14	22	43	14	27	51
7075	13.5	17	29	13.5	23	35	13.5	28	41
356	14	13	25.5	14	17.5	29.5	14	22	34

NOTE: (6) When hard anodic coatings are produced by dc power only, the resultant coating is not as hard and brittle or as wear resistant as the coating produced by the superimposition of ac power onto the dc power and by the attendant higher current densities.

NOTE: (7) When coating thickness measures the desired value, reduce the voltage to zero and remove parts immediately from the electrolyte.

NOTE: (8) Because metal is consumed in anodizing, the increase in diameter or thickness of a part due to the anodic coating gives a net growth of approximately one-half of the anodic thickness.

NOTE: (9) Where threads, etc., are not to be anodized, these surfaces shall be masked. After anodizing the required areas, remove the masking and re-anodize the threads or other area. Stop at 0.0002 to 0.0003 inch to avoid dimensional conflict and to provide corrosion protection.

3.9.3.4 Rinse

Parts shall be rinsed in cold water spray or by immersion in cold water. Do not use hot water. If parts are not masked, they should be sealed immediately. If parts are masked, remove the masking and re-rinse as masking may entrap electrolyte.

3.9.3.5 Low-density coating

When masked areas are desired to have a measure of anodic protection, the parts, after stripping the masking, shall be anodized as required and rinsed in cold water.

3.9.3.6 Seal

Components shall be sealed by immersion in the hard-anodize sealing solution given in paragraph 3.4.5.3 of this specification for 4 minutes.

3.9.3.7 Drain and rinse

Parts shall be drained when removed from the sealing bath and rinsed with sprayed cold water or immersed in flowing cold water.

3.9.3.8 Dry

Parts may be blasted with pressurized, filtered air or dried in still air. Do not oven dry.

3.9.4 Type IV colored anodizing

3.9.4.1 Limitations

The basic, anodic coating produced by Type I anodizing tends to give a muddy color when it is dyed, due to the inherent yellow coloring of the coating produced by chromic acid. For this reason, Type II anodic coatings, which are clear before dyeing, are preferred as a base. Alclad materials are difficult to color, require special handling, and should not be used without consent of the Initiating Activity.

Extruded 7075 alloys cannot be used because they exhibit surface flaws caused by extruding techniques.

All details and butting parts, classified as appearance items, shall be fabricated from the same alloy. All details of an assembly shall be fabricated so the grain of the material runs in the same direction as the length of the piece.

Sandblasting, grit-blasting, or vapor blasting on decorative parts prior to anodizing must be approved by the Initiating Activity.

3.9.4.2 Color processing

Parts that have been anodized and rinsed shall be immersed in agitated coloring baths of the dye concentration, pH and bath temperature given in paragraph 3.4.4 of this specification. The time of immersion shall be determined by test panels and manufacturer's recommendations.

3.9.4.3 Rinse

The parts shall be rinsed in cold, demineralized water.

3.9.4.4 Color seal

Colored parts shall be sealed by immersing in one of the baths listed in paragraph 3.4.5.4 of this specification. The particulars for the bath chosen must be operative.

3.9.4.5 Rinse

Parts shall be given a final hot rinse in 180 to 210° F water for a minimum of 5 minutes.

3.9.4.6 Dry

Parts shall be dried by blasting with filtered air or by drying in an oven not over 215° F.

4. QUALITY ASSURANCE PROVISIONS

4.1 Finish

The anodic coatings shall be continuous, adherent, non-powdery, free from clamp marks, burns, shading, or other damage. When no coating thickness is specified on the drawing, Type I, II, and IV shall be controlled by the weight of the coating as outlined in paragraph 4.2 of this specification.

4.2 Coating Weight

The minimum coating weights shall be:

Milligrams/Square Foot

	Non-Dyed	Dyed
Type I	200	500
Type II	600	2,500

Coating weight shall be determined per paragraph 4.4.3.2 of Specification MIL-A-8625A.

4.3 Corrosion - Resistance

The corrosion resistance shall be determined by the method outlined in paragraph 4.4.4.1 and 4.4.4.2 of Specification MIL-A-8625A.

4.4 Solution Maintenance

4.4.1 Anodizing solutions

4.4.1.1 Type I

In addition to the content of paragraphs 3.4.1 and 3.9.1.3 of this specification, the solution shall be analyzed when in regular use and as often as it is found necessary to control the following maximum contents:

- Chloride - determined as Na Cl - 0.2 gram per liter, max.
- Sulphate - determined as H_2SO_4 - 0.5 gram per liter, max.
- pH control shall be maintained by adding chromic acid until 10 percent (free + combined chromic acid) is present or 5.0 percent of free chromic acid as a minimum.

4.4.1.2 Type II

The sulphuric acid concentration shall be maintained at 165 to 175 grams per liter, and the aluminum content shall not exceed 20 grams per liter.

4.4.1.3 Type III

In addition to the content of paragraph 3.4.3, the solution shall be analyzed as often as it is necessary to maintain the alumina content at 10 grams per liter maximum. The specific gravity of the bath when tested at 75°F and at 10 grams of AL_2O_3 will be 1.137.

4.5 Stripping solution

When the acidic solution given in paragraph 3.5.4.2.B of this specification contains 10 grams of dissolved alumina per liter, it shall be discarded.

4.6 Sampling

The sampling procedures outlined in Specification MIL-A-8625A shall apply; however, for colored anodized parts, test panels of 6061-T6 may be approved as color chips.

4.7 Color Defects

Chromic acid anodized parts shall have no color standard and shall not be rejectable on the basis of color.

Lap marks due to double dips necessitated by length and size of parts are rejectable.

4.8 Coating Testing

Whenever coatings are stripped prior to welding or whenever coating coverage is in question, an electrical means of measuring conductivity shall be employed to verify the existence or non-existence of an anodic coating.

Type III anodic coatings shall be file hard when thicker than 0.0005 inch.

5. PREPARATION FOR DELIVERY

Not applicable to this specification.

6. NOTES

6.1 Intended Use

This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use in the anodizing of aluminum alloys for space launch vehicle applications.

6.2 Personal Safety

All necessary precautions regarding toxicity and industrial health hazards shall be taken by personnel engaged in anodizing in accordance with MSFC instructions and regulations. These precautions shall include adequate ventilation over tanks and safety in handling of chemicals, especially in the case of Type II or III anodizing baths. Suitable protective apparel shall be provided.

6.3 Chemical Storage

Chemicals shall be stored in cabinets or storage rooms which can be maintained in a clean and orderly condition and which can be easily flushed with water to wash away or to neutralize spillages.

6.4 Handling Containers

Containers used in the transfer of chemicals shall be of materials not easily corroded or attacked. They shall be thoroughly washed and cleaned after usage.

6.5 Records

Log sheets recording solution analyses, additions, temperatures, pH values, and age of the solution shall be kept on etching, anodizing, sealing, and dyeing tanks.

NOTICE: When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodians

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING SPECIFICATION FOR
THE APPLICATION OF CONVERSION COATINGS TO
ALUMINUM ALLOYS

1. SCOPE

1.1 Scope — This manufacturing specification covers the approved PE Laboratory manufacturing requirements for the application of conversion coatings to aluminum alloys.

1.2 Applicability — Conversion coatings covered herein are to augment paint adhesion and to provide a measure of corrosion protection for the following aluminum alloy types, clad or unclad; — 2014, 2024, 2219, 6061, and 7075.

2. APPLICABLE DOCUMENTS

2.1 Governmental — The following documents of the issue in effect on the date of use of this specification form a part of this specification.

SPECIFICATIONS

Federal

0-C-275 Chemicals, Photographic "Single Substance", General Specification For

0-T-634A Trichloroethylene, Technical

Military

JAN-A-183(2) Nitric Acid, 42° Baume'

MIL-C-5541 Chemical Films for Aluminum and Aluminum Alloys

MIL-S-13187(CmlC) Sodium Hydroxide, Technical

MIL-F-18264B(1) Finishes, Organic, Aircraft, Application and Control of

George C. Marshall Space Flight Center

MS 150.0

Manufacturing Specification for Vapor
Degreasing of Metallic Surfaces

STANDARDS

Federal

151A

Metals, Test Methods

2.2 Other Publications — The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

Amchem Products Inc.

Bulletin No. 624

Deoxidine (Deoxidizer)

(Copies of this publication may be obtained from Amchem Products Inc., St. Joseph, Missouri.)

Allied Research Products, Inc.

No. 14-2 Al-Coat
Bulletin

Operating Data for Iridite No. 14-2
Al-Coat for Aluminum and Aluminum
Alloys

(Copies of this publication may be obtained from Allied Research Products, Inc., 4404 E. Monument St., Baltimore 5, Maryland.)

Turco Products Inc.

Bulletin No. 78

Turco 4215 (Non-Silicated Alkaline Spray
Washing and Hot Tank Compound for Use
on Aluminum)

Bulletin No. 84

Turco Smut-Go No. 1 (Aluminum Deoxi-
dizer and Brightener)

(Copies of these publications may be obtained from Turco Products Inc., P. O. Box 1055, Wilmington, California.)

3. REQUIREMENTS

3.1 General — No deviation from the requirements of this manufacturing specification shall be permitted without prior written approval of the MSFC initiating activity.

3.2 Materials

3.2.1 Vapor - Degreasing solvent — Trichloroethylene or equal shall be used for vapor or liquid degreasing and shall conform to Specification O-T-634a, Type II.

3.2.2 Alkaline cleaner — The alkaline cleaner shall be a non-silicated type and shall not etch the base alloy or cladding. It shall be equal or equivalent to Turco 4215, when it is used at a concentration of 6 to 12 ounces per gallon, at a temperature of 140 to 180°F and at a pH of 8 to 10.

3.2.3 Deoxidizer — The deoxidizer employed shall be equal to Turco Smut-Go No. 1 when used at a concentration of 8 to 16 ounces per gallon of water, at ambient temperature and a pH of 2 to 4.

3.2.4 Demineralized water — Filtered water, possessing a minimum electrical resistivity of 50,000 ohms, shall be used for all rinsing and conversion coating solutions.

3.2.5 Conversion coating solution — For immersion, spray, or brush application, Iridite No. 14-2 Al-coat at a concentration of 1.25 to 4.0 ounces per gallon of aqueous solution with a pH of 1.2 to 1.7 at ambient temperatures shall be used. For spray applications the solution temperature may be increased to 120°F.

3.3 Equipment

3.3.1 General — The equipment used for the application of conversion coatings to aluminum alloys shall permit the attainment of all quality requirements of this specification.

3.3.2 Cleaning tanks — Tanks of suitable size, constructed of stainless steel or of hot rolled, S.A.E. 1020, plate steel which is protected by suitable coatings, may be used for cleaning parts. The welded construction shall include joints welded from inside and outside the tank.

3.3.3 Deoxidizer or conversion solution tank — Tanks of suitable size, constructed of stainless steel or of hot-rolled, S.A.E. 1020, plate steel covered inside with Koroseal, rubber, or other acid resistant coating and with joints welded inside and outside may be used.

3.3.4 Final rinse tanks — The rinsing tank immediately following the conversion coating tank may be constructed of hot rolled, S.A.E. 1020, plate steel with joints welded inside and outside. The inside surfaces may be protected with either Koroseal or painted with epoxy-resin paint.

3.3.5 Crane - An overhead, rail-type crane of sufficient capacity may service the tank area.

3.3.6 Racks

3.3.6.1 Cleaning — Racks for supporting parts during cleaning shall permit thorough draining and may be constructed of stainless steel or aluminum.

3.3.6.2 Conversion coating — Racks or perforated containers for use in conversion coating solutions may be constructed of aluminum alloys or titanium.

3.4 Preliminary cleaning

3.4.1 General — Surfaces to be conversion coated must be uniformly and thoroughly cleaned prior to application of conversion coatings. Cleaning is usually accomplished in three stages.

3.4.1.1 Solvent cleaning — Parts shall be vapor degreased per Specification MS 150.0 to remove all organic contamination and loosely held foreign matter embedded in the grease films.

3.4.1.2 Alkaline cleaning — Parts on which etching is objectionable shall be cleaned in a hot alkaline cleaner such as Turco 4215, or equivalent, to remove the last traces of organic contamination.

A. Rinse — When thoroughly cleaned, the parts after rinsing in water following this operation will exhibit no water-break.

B. Alkaline cleaning, optional — When etching is not objectionable or when aluminum castings are processed, a hot alkali-etch may be used. The usual concentration is 1 to 5 percent of sodium hydroxide and a bath temperature of 200° F maximum is used. Immersion time may vary from 15 to 60

seconds. This treatment serves as an alkaline cleaning and a deoxidizing treatment. If smut forms as a result of the hot alkali treatment, it shall be removed by immersing in a solution of Turco Smut-Go per paragraph 3.4.1.3.

C. Rinse — Parts subjected to the alkali-etch shall be thoroughly rinsed in cold water.

3.4.1.3 Deoxidizer — To produce a chemically cleaned surface, free from chips, adherent metal, smut and oxide, parts shall be treated with Turco Smut-Go No. 1 or equivalent for 5 to 10 minutes at the concentration and temperature in paragraph 3.2.3.

3.4.1.4 Rinse — Parts shall be rinsed in cold, flowing water to prevent deoxidizer or de-smutting chemicals being carried into the conversion bath.

3.5 Conversion Coating Procedure

3.5.1 Loading — Arrange parts to be coated by immersion so that they will permit the best drainage.

3.5.2 Iridite No. 14-2, immersion application — Parts shall be immersed in Iridite No. 14-2 (or equal) for 1/2 to 3 minutes. The bath shall contain 1.5 to 2.25 ounces of Iridite No. 14-2 compound per gallon of water, and its temperature shall be between 75 to 95°F. The pH shall be between 1.2 and 1.7.

3.5.3 Iridite No. 14-2, spray application — Parts shall be sprayed at a pressure of 5 to 50 psi with a solution containing 1.25 to 2 ounces of Iridite No. 14-2 compound (or equal) per gallon of water for a period of 15 seconds to 3 minutes. The solution temperature shall be between 70 to 120°F. The pH shall be between 1.2 and 1.7.

NOTE: All nozzles and other equipment in contact with Iridite No. 14-2 solution must be acid resistant.

3.5.4 Iridite No. 14-2, brush application — Brush parts with Iridite No. 14-2 solution (or equal) containing 4 ounces of Iridite No. 14-2 compound per gallon of water and at a solution temperature of 75 to 95°F. A single application is equivalent to a 5- to 10-second immersion treatment. Repeated applications of fresh solution will increase the film thickness and increase the protective value of the coating.

3.5.5 Cold rinse — Parts treated with conversion coating solution shall be rinsed in demineralized water at room temperature by spraying or in a tank which provides a flowing rinse.

3.5.6 Drying

3.5.6.1 Air drying — Parts may be dried by using pressurized air.

3.5.6.2 Oven drying — If oven drying is employed, the oven temperature must not exceed 150°F to avoid powdery coatings.

3.6 Stripping Conversion Coatings for Reprocessing — Parts shall be alkaline cleaned per paragraph 3.4.1.2 until no water-break is present after rinsing. It is permissible to immerse the part in deoxidizer to achieve this freedom from water-break. Subsequently, the part shall be immersed in a non-etching stripping solution until gassing begins. The stripping solution shall be at room temperature and composed as follows:

Nitric acid (40° Baume ^f)	3 parts by volume
Water	1 part by volume
Ammonium acid fluoride (Bifluoride)	7½ grams per gallon of solution

After stripping, the parts shall be thoroughly rinsed and recoated per paragraphs 3.5.1 to 3.5.7.

4. MANUFACTURING CONTROL PROVISIONS

4.1 Visual Examination — Coatings usually exhibit an iridescent color ranging from light golden to brown. The coating shall be continuous, smooth, adherent, and uniform in appearance.

4.2 Salt Spray Test — Conversion coatings when tested per Federal Standard 151A shall withstand 168 hours exposure (MIL-C-5541).

4.3 pH Determination — All determinations shall be made electrometrically using a potentiometer with a glass electrode and as often as it is necessary to maintain the pH ranges given in paragraphs 3.2.5.1 to 3.2.5.7.

4.4 Solution Control — When the conversion coating acquires a greyish cast instead of the usual yellow, the bath shall be analyzed for hexavalent chromium content. If after a corrective addition of Iridite No. 14-2 compound or equal, the pH is out of range, nitric acid (42° Baume') shall be added for lowering the pH. Whenever corrective additions are made, the bath should be stirred thoroughly.

4.4.1 Hexavalent chromium determination

Equipment: Pipette 10 ml
 Burette 50 ml
 Beaker 400 ml
 Graduate 10 ml
 Stirring rod

Reagents: All reagents used in this determination shall meet Specification O-C-275.

Solutions: 1. Sodium thiosulphate, $\text{Na}_2\text{S}_2\text{O}_3$, 0.1N
 Standardized against $\text{K}_2\text{Cr}_2\text{O}_7$
 2. Potassium iodide, KI, 10 percent
 3. Starch or thyodene indicator

Method: 1. Pipette 10 ml of solution into beaker and dilute to 250 ml with distilled water.
 2. Add 10 ml of potassium iodide and 5 ml of concentrated sulfuric acid. Stir.
 3. Titrate to a light yellow color using sodium thiosulfate solution.
 4. Add 1 to 2 ml of starch or thyodene solution.
 5. Continue titration with thiosulfate solution dropwise with constant stirring until blue color changes to a clear solution.

Calculations: $\text{Ml thiosulfate} \times \text{Normality} \times 0.81 = \text{oz Compound/Gal.}$

4.5 Cleanliness — The cleanliness of cleaned surfaces prior to conversion coating shall be checked by employing the water-break test outlined in paragraph 4.5.1.

4.5.1 Water-break test procedure — A mist of distilled water shall be atomized onto the surface under test, representative of the larger surface being tested, by employing any convenient small atomizing device. If the water gathers into discrete droplets within 25 seconds, and if the surface shows a water-break within that time, then the surface shall be considered as having

failed the test. If the water forms a continuous film but flashes out suddenly over a large area, then this characteristic shall presume the presence of free alkali, residual detergent or uncleanness, and the surface shall be considered as also having failed the test. If the water droplets coalesce into a continuous film of water without a sudden flash out and form a lens, then the surface shall be considered as having satisfactorily passed the water-break test.

5. PREPARATION FOR DELIVERY.

5.1 General — The protection given parts coated with conversion coatings during handling or shipment of finished parts must be ample and adequate to insure acceptable parts.

When parts for LOX service are involved, they shall be treated as required by purchasing agreements, drawings, or related specifications.

6. NOTES

6.1 Intended Use — This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for the application of conversion coatings to aluminum components of space launch vehicles.

6.2 Caution — Because of the strongly oxidizing nature of conversion coating chemicals, string, wood, paint, or organic material should not be placed in conversion coating solutions. Avoid contact with skin or clothing. The use of gloves and goggles is recommended.

NOTICE — When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MS 550.0

MANUFACTURING SPECIFICATION FOR
CHEMICAL MILLING OF ALUMINUM ALLOYS

1. SCOPE

1.1 Scope – This specification covers the approved PE Laboratory manufacturing requirements for the chemical milling of aluminum alloys to be used in space launch vehicle applications.

1.2 Applicability – The requirements set forth in this specification are applicable to the following unclad aluminum alloys:

- | | | |
|--------|--------|--------|
| ● 2014 | ● 5052 | ● 6061 |
| ● 2024 | ● 5086 | ● 7075 |
| ● 2219 | ● 5456 | ● 7178 |

2. APPLICABLE DOCUMENTS

2.1 Governmental – The following documents, of the issue in effect on the date of use of this specification, form a part of this specification to the extent herein indicated.

SPECIFICATIONS

Federal

O-A-51C	Acetone
O-T-634A	Trichloroethylene, Technical
TT-X-916	Xylene (For Use in Organic Coatings)

Military

JAN-T-171(2)	Toluene
JAN-A-183(2)	Nitric Acid, 42° Baume
MIL-G-3866A	Gloves, Cloth, Cotton, Knitted, Lightweight
MIL-G-21888	Gloves, Nylon, Anticontamination

George C. Marshall Space Flight Center

MS 150.0	Manufacturing Specification for Vapor Degreasing Metallic Surfaces
MS 150.3	Manufacturing Specification for the Application of Conversion Coatings to Aluminum Alloys

2.2 Other Publications - The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

Eastman Kodak Company

Bulletin Q-24	Kodak Photo-Resist
Bulletin P-36	Kodak Metal-Etch Resist

(Copies of these publications may be obtained from Eastman Kodak Company, Rochester 4, N. Y.)

Nordson Corporation

Instruction Manual	Airless Spray Coating Systems
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(Copies of this publication may be obtained from Nordson Corporation, 1000 Franklin Ave., Amherst, Ohio.)

Products Research Company

Technical Data PR-785 & PR-789
Sheet 1 - '60

(Copies of this publication may be obtained from Products Research Company, 3126 Los Feliz Blvd., Los Angeles 39, California.)

Wyandotte Chemical Corporation

Bulletin S-2719 Wyandotte 2487B

Etchant Bulletin Wyandotte MIL-ETCH
F-2441

(Copies of these publications may be obtained from Wyandotte Chemical Corporation, Wyandotte, Michigan.)

3. REQUIREMENTS

3.1 General - No deviation from the requirements of this manufacturing specification shall be permitted without prior written approval of the MSFC initiating activity.

3.2 Materials

3.2.1 Wiping Solvent - Acetone for removing primer or marking inks shall conform to Specification O-A-51C.

3.2.2 Degreasing Solvent - Trichloroethylene shall be used for liquid or vapor degreasing and shall conform to Specification O-T-634A, Type I for liquid degreasing and Type II for vapor degreasing.

3.2.3 Maskants - While neoprene-base paint is widely used, vinyl plastic or other formulations yielding a sharp, uniform line between masked and unmasked areas and possessing sufficient strength to resist collapsing when extending beyond already etched-out material, shall be equally acceptable. The maskant must be easily removed by hand stripping. PR-785 is a suitable material.

3.2.3.1 Masking Tape - Tape employed in masking shall be preferably lead-foil tape of approximately 0.008-inch thickness and shall be equal to Scotch No. 40 electroplaters tape (Minnesota Mining and Manufacturing Company).

3.2.4 Etchant - The solution used for etching shall produce a smoothly etched surface free from pits and from other evidence of non-uniform etching. The etchant shall be equivalent to Wyandotte MIL-ETCH and shall contain a minimum of 65 percent of sodium oxide. The use of chelating agents or seeding additives is permissible if no adverse effects to parts result in the etching process.

3.2.5 De-smutting solution - An aqueous solution, equal or equivalent to Wyandotte 2487B, for removing the smut formed while etching aluminum shall be operated at room temperature and at an equivalent concentration of 16 ounces of chromic acid per gallon.

3.2.6 Chem-milled components - All component surfaces to be etched shall be smooth, free of scratches, burrs or other defects conducive to uneven etching.

3.3 Equipment

3.3.1 General - The equipment used in the preparation and chemical milling of aluminum shall be suitable and adequate for obtaining the quality of etching required.

3.3.2 Maskant application equipment -

3.3.2.1 Spray coating - Nordson airless hot spray equipment used at 160° F, with air pressure of 80 psi and a hydraulic pressure of 800 psi or the Ransburg electrostatic spraying equipment, or equals, are necessary to provide a smooth, pinhole-free coating. A spray booth utilizing a water curtain is a necessary piece of equipment.

3.3.2.2 Roller coating - Rollers of the paint roller type, may be used on flat or convex surfaces. Pressurized rollers may be used if their performance is satisfactory.

3.3.2.3 Brush coating - Paint brushes may be used on large surfaces or for touch-up.

3.3.3 Gloves - Nylon or cotton gloves should be used for handling cleaned parts prior to masking.

3.3.4 Scribing knives - Thin-bladed knives, such as X-acto No. 1 with No. 11 or No. 16 blades, or a dissecting scalpel such as Harshaw H-19660 with H-19670 type blade or equivalent, are recommended for scribing masked parts.

3.3.5 Templates - Templates may be made of tempered sheet steel, of a light metal, or of laminations of glass cloth embedded in epoxy plastic. Templates shall be suitable for scribing the required pattern for inspecting the finished etching. They shall be of approximately the same size and contour as the part. The scribing edges should be smooth and perpendicular to the masked surface.

3.4 General Procedure

3.4.1 Preparation of solutions

3.4.1.1 Aqueous solutions - Tanks shall be filled approximately one-half full with tap water before adding the required chemicals. After adding the required chemicals, the bath shall be agitated until all chemicals are dissolved then fill the tank to full volume.

3.4.1.2 Maskants - If maskants as received are of heavier viscosity than required for effective coating, they shall be thinned by adding the recommended thinner to the concentrate in the required proportions to obtain the required consistency. If the maskant must be used at higher than room temperature, it shall be used at the required temperature.

3.4.2 Cleaning of components and surface preparation

3.4.2.1 General - To effect uniform etching or adhesion of the organic maskant, components must be thoroughly clean of oil, grease, ink, primer, scale, or other foreign matter.

3.4.2.2 Pre-cleaning - Components shall be cleaned of visible oil, grease, marking inks, forming lubricants, primer, or other contaminants by use of clean cloths or aluminum wool saturated with acetone or Type I trichloroethylene.

3.4.2.3 Degreasing - All components shall be degreased in the manner outlined in Specification MS 150.0.

3.4.2.4 Deoxidizing - Parts to be masked shall be deoxidized by immersing in an aqueous solution of Wyandotte MIL-ETCH at a concentration of 12 ounces per gallon and at a temperature of 160° F. Immersion shall continue until uniform gassing results on the surface of the part.

3.4.2.5 Rinse - Parts shall be immersed in cold flowing water and spray rinsed upon removal from the rinse tank.

3.4.2.6 Desmut - Parts shall be desmuted by immersing for a maximum of 1 minute in a solution of the concentration and at the temperature specified in paragraph 3.2.5.

3.4.2.7 Rinse - All parts shall be rinsed in cold, flowing water and examined for "water-break." If parts exhibit a "water-break," repeat the processing of paragraphs 3.4.2.2 through 3.4.2.7.

3.4.2.8 Drying - Parts to be masked shall be dried in an oven at 140 to 200° F or allowed to evaporate to dryness in clean, dust-free areas. After drying, parts shall be blown off with filtered, oil-free air to assure clean surfaces for masking.

3.4.3 Masking of components

3.4.3.1 General - All liquid maskants shall be thoroughly mixed and brought to operating temperature prior to application.

3.4.3.2 Thickness - The thickness of maskant desired is 5 to 8 mils for etching up to $\frac{1}{4}$ inch in depth, and 8 to 10 mils for etching over $\frac{1}{4}$ inch in depth. The maskant thickness is to be measured by a micrometer at a number of locations on the part. The first two coats shall be applied in 1 day to avoid collecting dust and foreign matter that might cause separation of coats.

3.4.3.3 Alternate maskant - Electroplated copper is acceptable as a maskant on small thin work. The copper can be deposited after a plating-resist is applied through a silk-screen stencil. After etching, the copper must be removed by a nitric acid dip.

3.4.3.4 Properties of organic maskants - The properties of a typical maskant are listed in Table 4-4.

TABLE 4-4. MASK CONCENTRATE PR-785
(Diluent - Toluene or Xylene)

<u>Brush or Roller Application Viscosities</u>	
1. Prime Coat	Zahn G - 2 cup, 20 (± 3) seconds, at 68° F
2. Final Coats	Zahn G - 2 cup, 90 (± 5) seconds, at 68° F

Spray Application Viscosity

1. All Spray Coats Zahn G - 2 cup, 20 to 30 seconds, at 68° F

3.4.3.5 Maskant application methods

A. Brush and roller method - Apply two coats of maskant, diluted per Table 4-4, with brush or paint roller. Care shall be exercised to minimize working the maskant. After maskant has dried to a tack-free state, repeat the application until required thickness is obtained.

B. Spray method - Maskant may be sprayed from equipment specified in paragraph 3.3.2.1. The spray nozzle shall be held 6 to 8 inches from the work. Allow parts to air dry to a tack-free condition before repeating the spraying to obtain the required coating thickness.

3.4.3.6 Oven curing - Masked parts may be oven cured instead of air dried by placing them in a recirculating air oven operating at 200 to 230° F for 1 $\frac{1}{4}$ hours.

NOTE: Upon removal from the oven, parts must be allowed to cool to room temperature without touching anything to avoid damage to or sticking of the maskant.

Parts may be immersed and cooled in cold water, if the water does not adversely affect the maskant adhesion.

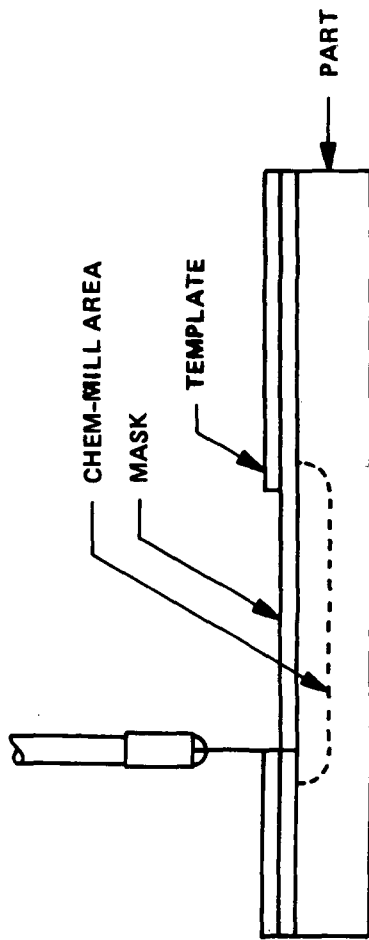
3.4.3.7 Scribing templates or silk-screens

A. Silk screens - When complex parts would require extensive scribing, silk screens may be used for applying maskant to the areas not to be etched.

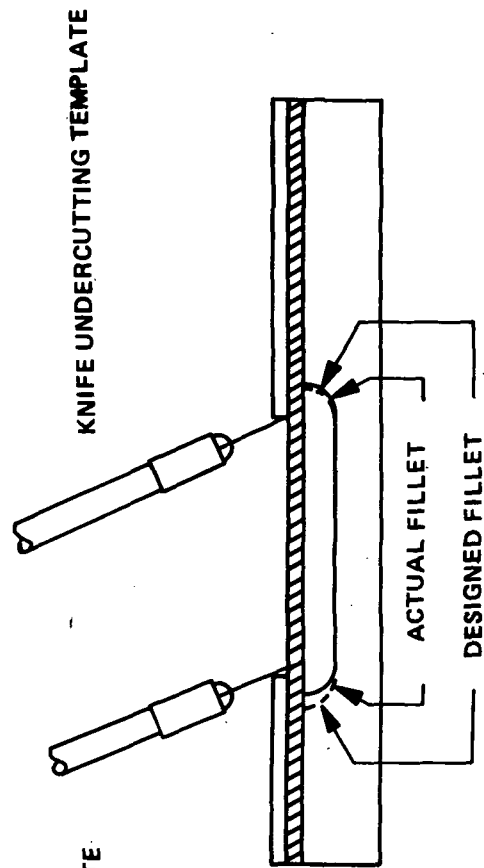
B. Other screens - When intricate detail on small, thin parts exists, the use of Kodak Photo-resist is permitted.

C. Templates - Templates shall be placed over the masked part, correctly located, then clamped against the part. Scribing shall be performed by placing the flat side of the knife against the edge of the cut-out area in the template and holding it perpendicular to the surface of the part. The blade shall be drawn along the edge of the template while exercising great care to avoid gouging the metal. Excessive pressure that mars the metal surface, thus permitting the etchant to reproduce the damage, may be cause for rejection of highly stressed parts. Figure 4-18 depicts correct and incorrect scribing. Figure 4-19 illustrates scribing damage reproduction. When scribing is complete, the template should be carefully removed.

BLADE PERPENDICULAR TO PART SURFACE AND PENETRATING MASK



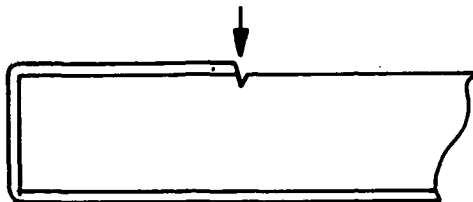
CORRECT METHOD OF SCRIBING



INCORRECT METHODS OF SCRIBING

Figure 4-18. Correct and incorrect methods of scribing.

KNIFE CUT OR SCRATCH RESULTING
FROM EXCESSIVE BLADE PRESSURE



SAME POINT
AFTER ETCHING

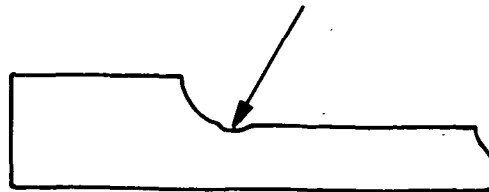
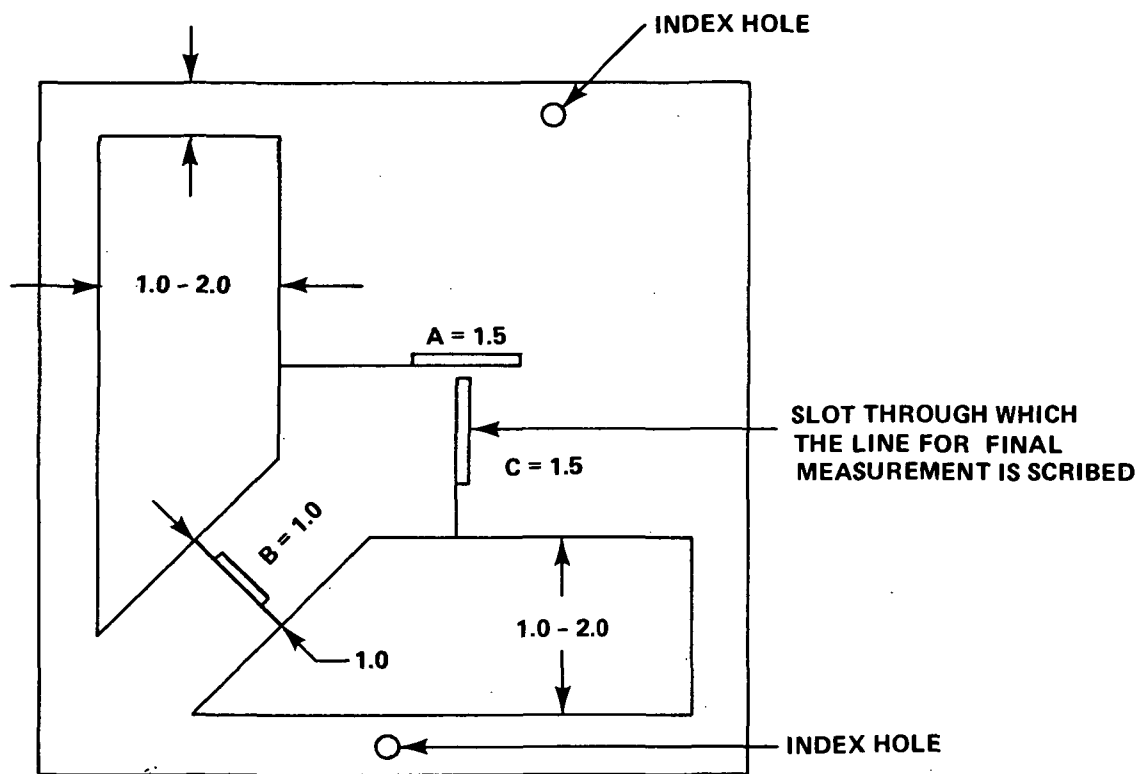


Figure 4-19. Scribing damage.



DISTANCES A, B, AND C SHOULD BE MEASURED AND STAMPED
ON THE TEMPLATE FOR READY REFERENCE

Figure 4-20. Template for etch factor determination.

3.4.3.8 Removal of scribed maskant - A sharp wooden instrument shall be used to lift a corner of the scribed maskant from areas to be etched. Maskant that is removed shall be peeled off by working towards the center of the cut-out areas. After stripping, any residual maskant must be removed by the wooden tool or an appropriate solvent. The remaining maskant shall be inspected for accuracy of pattern and obvious adherence to the base metal.

3.4.3.9 Maskant repair - Damage to the maskant, which will result in defective etched parts, must be repaired or the maskant must be stripped off completely. Damaged areas may be repaired by lead-foil tape firmly pressed in place, and edges of cut-outs may be repaired by localized spraying or brush applications of maskant.

3.4.4 Etch factor - Since the etchant removes metal in all directions, the etchant undercuts the edge of cut-outs approximately at the same rate as it removes metal perpendicularly to the exposed surface. In template layout, etch factors are used to determine the template overlap needed to produce the desired dimensions.

3.4.4.1 Etch factor determination - Etch factors for different alloy types, heat treatments, direction of grain, and etchant condition shall be determined by etching test panels such as illustrated in Figure 4-20. The procedure for determining etch factors is as follows:

- Clean and mask the panels that have been cut to size and drilled for locating onto template.
- Locate panel under template and clamp the two together.
- Using template as a guide, scribe the required pattern.
- Remove template and carefully peel off unwanted maskant.
- Etch panel as required.
- Rinse, desmut, demask, and dry panel.
- Apply template and scribe a light reference line through slots provided in template.

- Measure distance between etched edges at the proper points.
- Calculate etch factors as shown in Figure 4-21 for dimensions A, B, and C in Figure 4-20.

3.4.5 Etching - Using a concentrated etchant meeting the requirements of paragraph 3.2.4 and in the proportions required to obtain an etching rate of 1 mil per minute at 190° F ($\pm 5^\circ$), make up the etching solution as recommended in paragraph 3.4.1.1.

3.4.5.1 Loading of parts - Small parts shall be suspended in the etchant by racks or in baskets. Larger parts shall be suspended or supported as required.

3.4.5.2 Rotation of parts - It is sometimes necessary to rotate parts while in the etchant or to turn parts end-for-end to obtain uniform etching. If parts are rotated, the speed shall be controlled.

3.4.5.3 Taper etching - Tapers produced by either slowly lowering a part into or slowly extracting a part from the etchant must be controlled to obtain accurate configurations. It is recommended that the last cycle should be immersion followed by rapid withdrawal to alleviate smutting.

3.4.5.4 Immersion - Parts shall be allowed to remain in the etchant for the time required by the etch rate, and shall be inspected for cut depth after rinsing. If it is necessary, return parts to the etchant until the required cut depth is achieved.

3.4.5.5 Rinse - Parts shall be rinsed by immersion in cold flowing water then spray rinsed upon removal from the rinse tank.

3.4.5.6 Desmutting (Brightening) - Smut shall be removed from the part by immersing the part in an aqueous solution of Wyandotte 2487B, or equivalent, maintained at an equivalent concentration of 16 ounces of chromic acid per gallon and at room temperature. Immersion time varies from 3 to 10 minutes.

3.4.5.7 Rinse - Parts shall be rinsed in cold, flowing water.

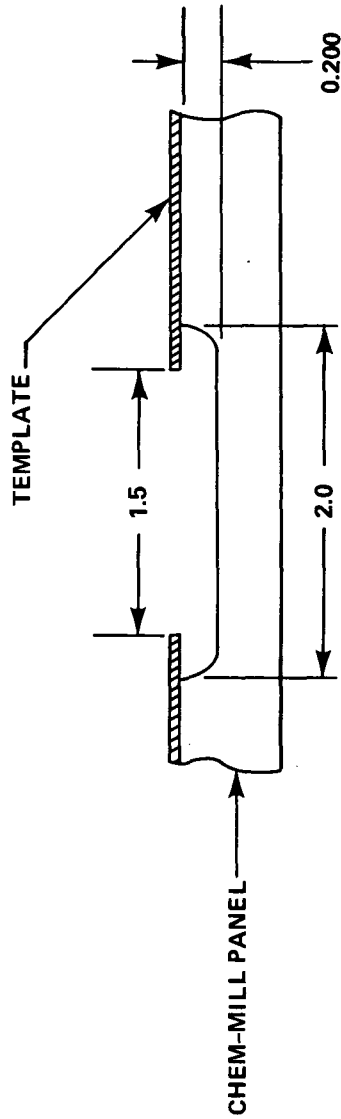
3.4.6 Demasking - Stripping of maskant shall be done by hand and parts shall be rinsed and inspected for retained maskant after drying.

CALCULATE THE ETCH FACTOR AS FOLLOWS:

$$F = \frac{W}{2 \times D} = \frac{\text{INCREASE IN MEASURED WIDTH}}{2 \times \text{DEPTH}}$$

FOR EXAMPLE:

THE TEMPLATE GAP, AS ILLUSTRATED BELOW, IS 1.5 IN. AFTER A 0.200 IN. CUT IS MADE, THE DISTANCE BETWEEN THE ETCHED LINES IS 2.0 IN.



$$F = \frac{2.0 - 1.5}{2 \times 0.200} = \frac{0.50}{0.40} = 1.25$$

Figure 4-21. Etch factor calculation.

4. QUALITY ASSURANCE PROVISIONS

4.1 Surface Finish - Roughness of etched surfaces parallel to the grain shall not exceed 125 micro-inches RMS and on the average shall be 50 - 90 micro-inches RMS. Unevenness of an edge due to mask imperfection shall not be considered as a pit but subject to rework.

4.2 Dimensions - Unless otherwise specified on engineering drawings or in the purchasing agreement, tolerances applicable to etched parts shall be:

- Lands - ± 0.020 inch
- Depths - ± 0.002 inch on light cuts; ± 0.005 inch on $1/8$ inch and deeper cuts
- Fillet radii - plus zero minus 20.0 percent
- Corner radii - plus or minus 20.0 percent

4.3 Edges - Sharp edges developed in etching shall be broken by a 0.005- to 0.030-inch radius and the radiused surface blended into adjacent surfaces. The technique employed shall not increase the susceptibility of the part to corrosion.

4.4 Reworked Parts - When parts have been etched to the limit in some areas but are defective in another, they may be corrected by machining methods if possible.

4.5 Solution Maintenance - The two major results of ignoring solution conditions are unacceptable finish and loss of lateral control. Etch factors change and templates become useless. Furthermore, without control of the sodium aluminate content the etching is pitty, rougher, uncontrolled, and under-cut varies or exceeds the cutting down rate.

4.5.1 Analysis of etching solution

4.5.1.1 Determination of aluminum content

A. Reagents and apparatus

- Copper powder
- 1 Normal sulphuric acid

- 1 - 100 cc beaker
- 1 - 5 cc pipette
- 1 - 25 cc burette
- 1 - 50 cc sample jar

B. Sampling

- Sample the well agitated bath
- Stopper the sample jar and allow sample to cool to 75° F
- Add 5 grams of copper powder and shake vigorously
- Allow solids to settle

C. Electrometric Titration

- Pipette 5 cc of the clear liquid into a 100 cc beaker, and add 25 cc of distilled water
- Titrate with 1N sulphuric acid until a pH of 12 is reached. Record the milliliters of acid as "N₁"
- Continue the titration with 1N sulphuric acid until a pH of 8.3 is reached. Record the increase in milliliters as "N₂"

4.5.1.2 Adjustment of concentrations - When the N₁ and N₂ values have been determined for a new bath, locate their intersection on a graph such as Figure 4-22 and position the oblongs so that point A is located analogously as it is in Figure 4-22. To increase N₁ by 1 milliliter (cc), add 1.06 ounces of etchant per gallon of solution. To increase N₂ by 1 milliliter, add the amount of sodium aluminate or scrap aluminum to maintain a minimum dissolved aluminum content of 0.16 pound per gallon. To determine the amount of dissolved aluminum in pounds per gallon, multiply the N₂ value by 0.045.

When the N₂ value has reached 0.75 (±0.05) pounds per gallon of dissolved aluminum, a portion of the bath (25 to 50 percent) should be discarded and the bath brought up to volume with fresh etchant.

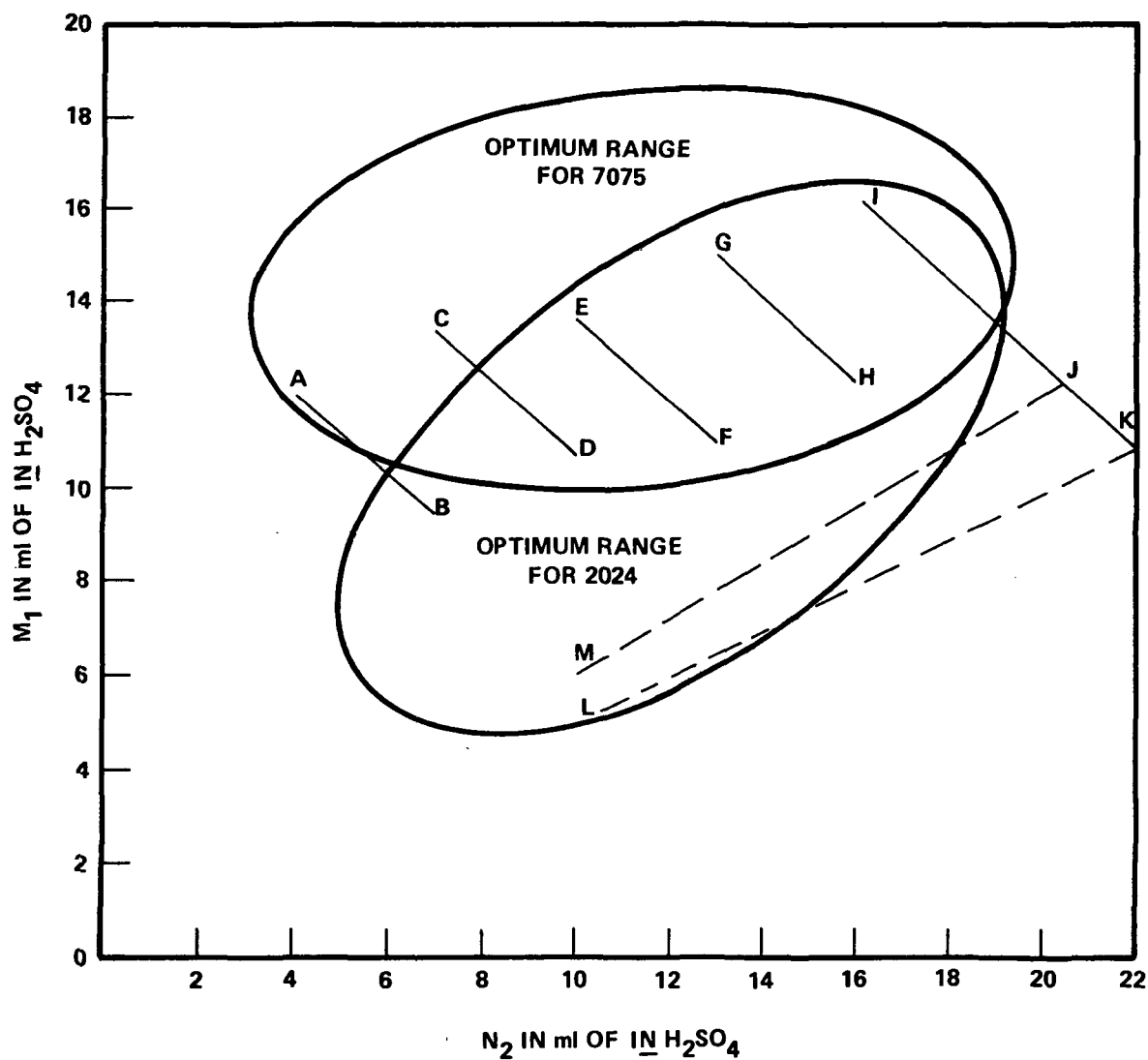


Figure 4-22. N_1 values versus N_2 values.

A. Example (1000 gallons of etchant) - A freshly prepared bath has a N_1 value of 12.0 and a N_2 value of 4.0, which are the coordinates of point "A" in Figure 4-22.

Parts are presumed to be etched until 120 pounds of aluminum have been dissolved and the bath analysis is N_1 equals 9.4 and N_2 equals 7.0. These are coordinates of point B. 254 pounds of etchant are added to raise N_1 to 13.4 (N_2 remains essentially unchanged). The restored bath is represented as point "C" of Figure 4-22.

When point I is reached, parts are etched until bath becomes inefficient. Point J represents the bath condition if 180 pounds of aluminum have dissolved. Point K represents the condition of the bath if 240 pounds of aluminum are dissolved. The bath may be rejuvenated by dumping 50 percent of the solution and restoring the volume with water. The concentrations would be represented by point L.

The value of N_1 is raised to point E by the addition of 570 pounds of etchant. The bath is now restored to operating volume with water and the whole cycle continued indefinitely.

The ratio of etchant to dissolved aluminum will vary from 3.5:1 to 2.5:1 depending on the point of dumping a portion of the solution on line I-K in Figure 4-22.

4.5.1.3 Zinc and copper content - When the dissolved zinc and copper content of the bath causes uneven etching, a portion of the bath may have to be dumped and the bath restored as outlined in paragraph 4.5.1.2.A.

4.6 Cleanliness - The cleanliness of cleaned surfaces prior to masking shall be checked by employing the water-break test outlined in paragraph 4.5.1 of Specification MS 150.3.

4.7 Maskant Adhesion - The maskant, when tested on air cured or oven cured test pieces, shall show a minimum adhesive strength of 1.5 pounds per lineal inch of width.

4.8 Thickness Measurement - Maskants or metal sections shall be measured by micrometer caliper or other direct reading means wherever possible. The use of an ultrasonic gaging means is permitted on metal parts that are inconvenient to measure directly.

5. PREPARATION FOR DELIVERY

5.1 General - The protection given chemically milled parts during handling or shipment of finished parts must be ample and adequate to insure acceptable parts.

6. NOTES

6.1 Intended Use - This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use in the chemical milling of aluminum alloys for use in space launch vehicle applications.

6.2 Safety Precautions - All necessary safety precautions regarding toxicity, skin contact, and industrial health hazards shall be taken by personnel engaged in chemical milling in accordance with MSFC instructions and regulations. Adequate ventilation shall be maintained over processing tanks and proper precautions taken to assure safe conditions in case of spillage, especially of the concentrated etchant. Suitable gloves, protective clothing, and devices shall be available.

All materials shall be stored in chemical store rooms which shall be maintained in a clean and orderly condition. The floors shall permit flushing with water to wash away spillages.

6.3 Handling Containers - Containers used in the transfer of chemicals shall be of materials not easily corroded or attacked. They shall be adequately cleaned after use.

6.4 Maskant Storage - Maskants shall be purchased in quantities that will result in a rapid turnover and will not permit the storage of a maskant for over 6 months. Maskants are preferably stored in a cool place.

NOTICE: When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodians:

Process Engineering Laboratory

George C. Marshall Space Flight Center

Preparing Activity:

Process Engineering Laboratory

George C. Marshall Space Flight Center

MANUFACTURING SPECIFICATION FOR CHEMICAL MILLING OF ALUMINUM HONEYCOMB

1. SCOPE

1.1 Scope - This specification covers approved PE Laboratory manufacturing requirements for chemical milling aluminum honeycomb cores of space launch vehicle components.

1.2 Applicability - The requirements of this specification are applicable to aluminum honeycomb fabricated of the following non-clad, aluminum alloys - 3003 H19, 5052 H39, and 5056 H39.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this specification, form a part of this specification to the extent indicated herein.

Federal

O-T-634a

Trichloroethylene, Technical Grade

Military

MIL-C-7438C(2)

Core Material, Aluminum, for Sandwich Construction

MIL-F-18264B (1)

Finishes, Organic, Aircraft, Application and Control of

George C. Marshall Space Flight Center

MS 150.0

Manufacturing Specification for Vapor Degreasing of Metallic Surfaces

MS 550.0

Manufacturing Specification for Chemical Milling of Aluminum alloys

2.2 Other Publications - The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

American Mineral Spirits Company

Bulletin 10 - ' 58

AMSCO Special Naphtholite P-1

(Copies of this publication may be obtained from American Mineral Spirits Company, Western 8600 S. Garfield Avenue, Box 551, South Gate, California.)

Daystrom Inc.

06-500, Nov. ' 59

Model 8009, Weston Midget Moisture
Meter

(Copies of this publication may be obtained from Daystrom Incorporated, Weston Instruments Division, 4866 Cooper Road, P. O. Box 42033, Cincinnati 42, Ohio.)

Turco Products Inc.

Bulletin No. 11 - ' 59

Honeycomb Chem-Mill Products

Bulletin No. 50

Aviation, Hot Tank Cleaner

Bulletin No. 165

Turco 3878

(Copies of these publications may be obtained from Turco Products Inc., P. O. Box 1055, Wilmington, California.)

United States Gypsum Company

Bulletin No. IGL-104

Ultracal 30

(Copies of this publication may be obtained from U. S. Gypsum Co., 300 W. Adams, Chicago, Illinois.)

Wyandotte Chemicals Corporation

Bulletin F-2441

Mil-Etch

(Copies of this publication may be obtained from Wyandotte Chemical Corporation, Wyandotte, Michigan.)

3. REQUIREMENTS

3.1 General - No deviation from the requirements of this manufacturing specification shall be permitted without prior written approval of the MSFC initiating activity.

3.2 Materials

3.2.1 Degreasing - Trichloroethylene, or equal, shall be used for vapor or liquid degreasing and shall conform to Specification O-T-634a.

3.2.2 Alkaline cleaner - Turco Aviation, or equivalent, at a concentration of 4 to 6 ounces per gallon and at 160 to 180° F shall be used for final degreasing.

3.2.3 Molding material

3.2.3.1 Matching plaster - The material used for forming the master mold shall be equivalent or equal to Turcoform Master Mold or Ultracal 30 plaster.

3.2.3.2 Plaster negative - The material which is used for forming the negative or reverse configuration present in the master mold, and which will permit embedding of the honeycomb without fragmentation, shall be equal or equivalent to Turcoform Negative.

3.2.4 Release material, No. 1 - The pliable film used as a separator between the matching plaster and the plaster negative shall be equal or similar to Turcoform Release No. 1.

3.2.5 Maskant - The maskant applied to the negative plaster shall be equal or equivalent to Turcoform Mask 510.

3.2.6 Release material, No. 2 - The material used over the maskant and adjacent to the Cor-Seal shall be equal or equivalent to Turcoform Release No. 2.

3.2.7 Core sealant - The material used to fill that portion of the honeycomb core not to be etched shall be equal to Turcoform Cor-Seal.

3.2.8 Etchant - The concentrated etchant used to dissolve the honeycomb which is not protected by the maskant shall be equal or equivalent to Wyandotte Mil-Etch.

3.2.9 Demaskant - The demasking bath shall be equivalent to Turcoform Demask 607 and shall either remove or loosen the maskant to ease the task of removing the maskant.

3.2.10 Aluminum honeycomb - Blank honeycomb shall conform to Specification MIL-C-7438C (2) or other specifications mentioned by drawings or purchasing agreements.

3.3 Equipment

3.3.1 General - The equipment used in the preparation and etching of aluminum honeycomb cores shall be suitable for obtaining accurately etched configurations.

3.3.2 Processing tank construction - The alkaline cleaner, rinse, and etching tanks shall be constructed of heavy gauge, hot rolled, S.A.E. 1020, steel plate with joints welded inside and outside and with sufficient insulation to retain the required temperature.

3.3.2.1 Special tank features

A. Fume exhaust - Tanks requiring venting shall have slotted vents located at the tank flange on three sides connected by a plenum chamber to a forced draft fan. The fourth side shall be equipped with a perforated, compressed air line which, in conjunction with the exhaust vents, will maintain an air-flow of 100 to 250 C. F. M. per square foot of tank area.

B. Heaters - The alkaline cleaner and etching tanks shall have steam coils or immersion electric heaters with thermostatic controls, so that temperatures will be held to $\pm 5^{\circ}$ F.

C. Rinsing - The rinse tanks should possess 12 inches of additional depth below other tanks to permit installation of water nozzles. Vee Jet or equivalent nozzles with foot or remote control shall permit either hot or cold rinse.

3.3.3 Drying or curing oven - A recirculating, unit-heater type oven capable of maintaining 400° F ($\pm 5^\circ$) shall be used for curing the negative or maskant.

3.3.4 Hydraulic press - A hydraulic press capable of exerting 100 psi on the area of the core at a controlled ram speed not exceeding 1/16th inch per minute shall be used for embedding the core into the Turcoform negative.

3.3.5 Moisture meter - A moisture meter capable of measuring the moisture content of the cured Turcoform negative without damaging the negative shall be equal or equivalent to the Midget Moisture Meter, Model 8009, manufactured by Daystrom Incorporated, Weston Instruments Division, Cincinnati, Ohio.

3.3.6 Miscellaneous equipment

3.3.6.1 Melting pot - A melting pot for plastics with automatic stirring is required for the liquefaction of the core sealant.

3.3.6.2 Wash rack - A wash area with suitable water spray equipment is required for removing the plaster negative from the honeycomb.

3.3.6.3 Temperature measurement - A copper-constantan thermocouple and a portable potentiometer shall be used for measuring the maskant temperature during curing.

3.3.6.4 Work tables - Because of the weight of the plaster match or negative, sturdy, flat tables are required.

3.3.6.5 Blower - A hot-air blower is desirable for eliminating bubbles in the Cor-Seal.

3.3.6.6 Gloves - Nylon or cotton gloves should be provided for handling cleaned honeycomb.

3.3.6.7 Brushes - Paint brushes of various sizes and shapes are used in applying maskant to the Turcoform negative.

3.3.6.8 Spray equipment - Spray equipment consisting of a spray gun equivalent to De Vilbiss, Type PMBC No. 510-2E with nozzle No. 24 and tip AV-15-D or Binks No. 7 spray gun with a 36 x 36 P kit, pressure pots, spray tables, and a water-curtain, spray booth shall be available for spraying maskant.

3.3.6.9 Polishing equipment - Light-weight hand tools equipped with fine-grit emery paper shall be used to remove surplus node material from etched honeycomb.

3.3.7 General procedure

3.3.7.1 Honeycomb cleaning - Prior to embedding in the negative, the core shall be clean of oil, grease, and other foreign matter to insure maskant adhesion and to promote uniform etching.

A. Degreasing - Cores may be precleaned with trichloroethylene in a standard vapor or liquid degreaser in the manner outlined in Specification MS 150.0.

B. Alkaline cleaning - Degreaser cleaned cores shall be alkaline cleaned in an aqueous solution of 4 to 6 ounces per gallon of Turco Aviation, or equivalent, at 160 to 180° F for 10 to 20 minutes, or until no evidence of a "water-break" is manifested after the following rinse operation.

C. Rinse - Cores that have been alkaline cleaned shall be spray rinsed as they enter or leave a hot rinse tank.

D. Dry - Cores shall be dried by pressurized air which has been filtered to remove water and oil.

3.3.7.2 Plaster mold casting

A. Master mold or match - Employing conventional pattern shop techniques and mixing the plaster or resin according to the producer's instructions, construct the master or match to reproduce the exact configuration desired in the honeycomb core. It must also have the excess material in the base shown in Figure 4-23 a (step 1). Side A is flat and oriented so that it is perpendicular to honeycomb cell walls in the finished part.

After the resin or plaster is cast and aged for 24 hours, it shall be used for the production of the plaster negative. The plaster master should be checked for dimensional accuracy before reusing if it has aged for more than 2 weeks.

B. Plaster or Turcoform negative - Using the master or match just produced as a base, place a sheet of Turcoform Release No. 1, or other successful separator, over the master to act as a separation between the master and the negative to be poured. In the case of sheeting, the vacuum bag technique

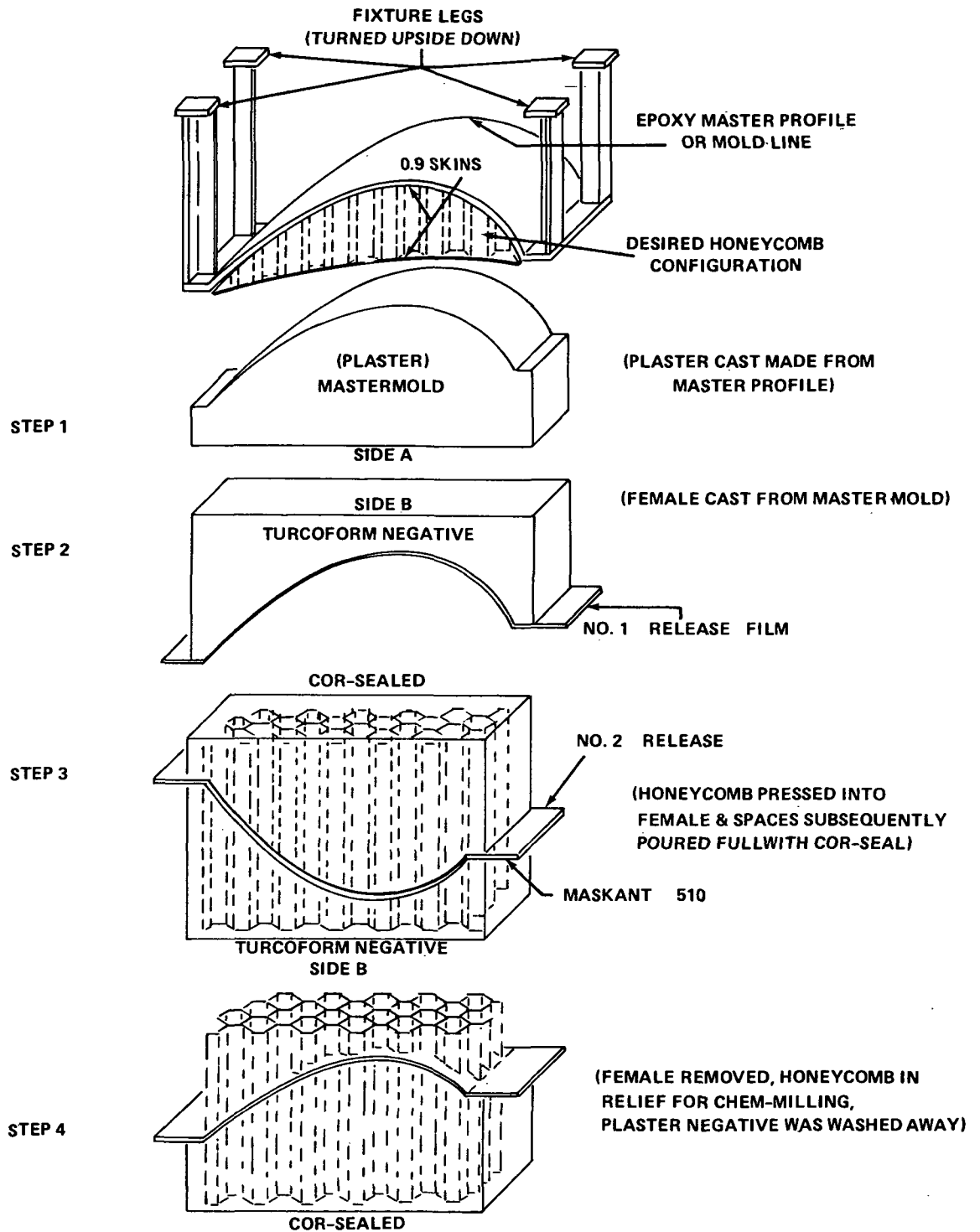


Figure 4-23 a. Chemical-milling aluminum honeycomb (steps required to produce top outside skin mold line)

achieves the necessary form-fitting contact with the master. Position a box frame on top of the master for retaining the liquid negative when poured. The box frame shall allow at least 0.5 inch space on four sides between it and the honeycomb core. The negatives, Figure 4-23, Step 2, Side B, shall be finished perfectly flat and parallel to Side A.

After the negative is poured and finished and allowed to set for 30 to 60 minutes, carefully invert the assembly and cautiously lift the master from the negative.

Turcoform negative must be oven cured at 120° F for 16 to 24 hours or until the retained moisture is reduced to 8 to 10 percent.

3.3.7.3 Masking - The negative shall be coated with maskant to a thickness of 10 to 15 mils. If applied by hand, Turcoform Mask 510 is diluted with Mask 510 additive in the ratio of four parts maskant to one part by weight of additive. If the maskant is sprayed, the maskant is further diluted with 10 percent by weight of Amsco Special Naphtholite or equivalent. After application, the maskant shall be cured at 180° F for 1 hour to reduce tackiness.

3.3.7.4 Release No. 2 application - Whenever the core height is 1.0 inch or more and the cell size 3/16 inch or less, Turcoform Release No. 2 shall be applied over the maskant to a dry film thickness of 6 to 10 mils. This release should be applied at least 15 minutes before core embedment into the negative.

3.3.7.5 Embedment of core - Locate the blank honeycomb core over the Turcoform negative, that has been masked and possibly coated with Release No. 2 or equivalent. Embed the core at a penetration rate not greater than 1/16 inch per minute until the pressure platen bears against rigid limiting stops.

3.3.7.6 Final curing of mask - After the core is embedded, the assembly shall be placed in a recirculating oven and held at temperature for 1 hour after the maskant reaches 325° F. A thermocouple inserted in the maskant should be used to measure the temperature.

3.3.7.7 Core sealing - After the core and negative assembly are removed from the oven and before it cools to room temperature, fill the honeycomb cells with Cor-Seal or equivalent. Cor-Seal shall be heated to 370° F ($\pm 5^\circ$) in a plastic melting pot before pouring. Any bubbles formed shall be allowed to escape by using a warm air blower.

The Cor-Seal shall be poured in a box frame which surrounds the honeycomb and which provides a clearance of 1/2 to 3/4 inch above and around the core.

Handling hooks or devices shall be embedded in the Cor-Seal before it congeals to accommodate racking or hold down devices used to offset the buoyancy of the Cor-Seal.

3.3.7.8 Negative - As soon as the Cor-Seal has congealed, the negative may be washed out of the honeycomb by a stream of water. It is not necessary to extract all of the negative as long as none of it masks the cell walls.

3.3.7.9 Etching - Honeycomb cores protected by Cor-Seal and maskant shall be etched per Specification MS 550.0. The buoyancy of the assembly must be overcome by weights or racking. The open mesh shall be on the top side during etching to permit hydrogen to escape easily.

3.3.7.10 Rinse - Cores that are removed from the etchant shall be thoroughly rinsed in cold water.

3.3.7.11 Core dressing - Since the bonding material at the nodes of aluminum honeycomb is unaffected by the etchant, it must be removed by light sanding with 100- to 180-grit emery cloth. Minnesota Mining and Manufacturing Company's PG wheel is a serviceable tool for general dressing.

3.3.7.12 Cor-Seal removal - If Release No. 2 has not been used, the mask will be bonded to the Cor-Seal and will be removed with it. If Release No. 2 has been used, the mask will remain in the honeycomb.

Cor-Seal can be removed by mechanically pulling it free of the core. Large sections of core are readily freed of Cor-Seal by rolling them over a 4-inch cylinder.

3.3.7.13 Mask removal - Any mask remaining in the core shall be loosened by immersing in Turcoform Demask 607 or equivalent for 10 to 30 minutes. The loosened mask may be blown out of the cells by pressurized air.

3.3.7.14 Final cleaning - All components are to be cleaned per paragraphs 3.3.7.1 to 3.3.7.1.C to the level of cleanliness required by the service of the component.

3.3.7.15 Top and bottom mold lines - When the honeycomb core does not have one flat side, the procedure given in paragraphs 3.3.7.2 to 3.3.7.14 must be performed for the opposite side of the core. In this case a form die possessing the shape of the first plaster negative would be used to embed the previously etched core into the second plaster negative. Cor-Seal would then cover the previously second plaster negative. Cor-Seal would then cover the previously etched configuration to retain its integrity.

4. MANUFACTURING CONTROL PROVISIONS

4.1 Honeycomb - All aluminum honeycomb blanks shall be inspected for conformance to Specification MIL-C-7438C (2) and part drawings prior to cleaning.

4.2 Water-Break Test - This manifestation is described in paragraph 5.1.4.2.1 of Specification MIL-F-18264B (1).

4.3 Dimensional Control - Masters or master pattern blocks composed of plaster or other material that changes with time shall be checked for accuracy if the interim between production exceeds 2 weeks.

4.4 Etch - The finished etched core shall be free of scallops at the junction of the core and the outside skin of the component. The etched core should also be checked for trueness by a fixture employing templates for the mold lines at various stations on the component.

5. PREPARATION FOR DELIVERY

5.1 Levels of Preservation and Packaging - The requirements for preservation and packaging given in MIL-C-7438C (2) shall govern in the absence of definition in the purchasing agreement.

6. NOTES

6.1 Intended Use - This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use in the chemical milling of aluminum honeycomb cores used in space launch vehicle components.

6.2 Safety Precautions

6.2.1 Personal safety - All necessary safety precautions regarding toxicity, skin contact, and industrial health hazards shall be taken by personnel engaged in chemical milling in accordance with MSFC instructions and regulations.

Adequate ventilation shall be maintained over the processing tanks, and proper precautions taken to assure safe conditions in the event of spillage of the etchant.

6.2.2 Chemical storage - Demask materials, thinners, maskants, core sealers, and release agents shall be stored in rooms maintained in a clean and orderly condition and which can easily be flushed with water to wash away or neutralize spillages.

6.2.3 Handling containers - Containers used in the transfer and blending of chemicals shall be of materials not easily corroded or attacked. They shall be adequately cleaned after use.

NOTICE - When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING PROCESS FOR
THE CHEMICAL MILLING OF BULKHEAD GORES
FOR
THE S-IC VEHICLE

1. SCOPE

1.1 Scope - This procedure covers approved PE Laboratory manufacturing requirements for the chemical milling of S-IC bulkhead gore segments.

1.2 Applicability - The chemical milling of bulkhead gore segments shall be accomplished by the procedures outlined in this document. This process applies only to 2219 aluminum alloy, either -T37 or -T87 temper.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this specification, form a part of this specification.

SPECIFICATIONS:

Federal

O-A-51C	Acetone
O-T-634	Trichloroethylene, Technical

Military

Mil G-3866A	Gloves, Cloth, Cotton, Knitted, Lightweight
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George C. Marshall Space Flight Center

M.S. 150.0	Manufacturing Specification for Vapor Degreasing of Metallic Surfaces
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2.2 Other Publications - The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

Products Research Company

Technical Data PR-785 and PR-789

Sheet 1 - 1960

(Copies of this publication may be obtained from Products Research Company, 3126 Los Feliz Blvd., Los Angeles 39, California.)

Wyandotte Chemical Corporation

Bulletin 5-2719 Wyandotte 2487-B. (Copies of this publication may be obtained from Wyandotte Chemical Corporation, Wyandotte, Michigan.)

Nordson Corporation

Instruction Manual - Airless Spray Coating Systems

(Copies of this publication may be obtained from Nordson Corporation, 1000 Franklin Avenue, Amherst, Ohio.)

3. REQUIREMENTS

3.1 Chemical Milling Operators - The chemical milling operators shall be skilled in the art of applying maskants, in the scribing of details on the masked part, and in the chemical milling of aluminum parts.

3.2 Equipment

3.2.1 Tanks - The chemical milling tank shall be constructed of unlined mild steel. The desmutting tank shall be constructed of mild steel with a Koroseal lining on interior surfaces. The rinse tank shall be constructed of mild steel with a Plastisol lining on interior surfaces. Alternate tanks may be used provided these are acceptable to the procuring activity. Exhaust fans and vented hoods are necessary for safeguarding the health of workers in the area.

3.2.2 Controls - Adequate controls are required to maintain the etchant solution at 195°F ($\pm 10^\circ$) depending on concentration. The temperature control shall be monitored by thermometer readings.

3.2.3 Holding Frame - A suitable holding frame is required to hold the gore segments in the cleaning, chemical milling, and desmutting solutions. The attachment shall be motorized so that the gore can be moved horizontally 3 to 4 inches from 1 to 20 times per minute to promote uniform milling action

on the gore in the chemical milling solution only. The holding frame shall be constructed of 304 stainless steel or other suitable material.

3.2.4 Vacuum Line - If the alternate masking procedure outlined in paragraph 3.3.6.2 is used, then a source of vacuum will be necessary to evacuate the area inside the neoprene boot to 22 inches of Hg.

3.2.5 Maskant Application Equipment

3.2.5.1 Spray Coatings - Nordson Airless Hot Spray Equipment used at 120° F ($\pm 20^\circ$) with air pressure of 80 psi and a hydraulic pressure of 800 psi, or approved alternate, is necessary to provide a smooth, pinhole-free coating. A large spray head such as 68CO9 should be used. A spray booth or some other suitable equipment is necessary for removing maskant from the air.

3.2.5.2 Roller Coatings - Rollers, of the paint roller type, may be used on flat or convex surfaces.

3.2.5.3 Brush Coating - Paint brushes may be used on large surfaces or for touch-up.

3.2.5.4 Neoprene Boot - A specially designed neoprene boot with vacuum attachment may be used.

3.2.6 Gloves - Nylon or cotton gloves must be used for handling cleaned parts prior to masking.

3.2.7 Scribing Knives - Thin-bladed knives, such as X-acto No. 1 with a No. 11 or No. 16 blade, or a dissecting scalpel such as Harshaw H-19660 with H-19670 blade, or equivalent, are recommended for use in the scribing of masked parts.

3.2.8 Templates - Templates may be made of laminations of glass cloth embedded in epoxy plastics. Templates shall be suitable for scribing the required pattern on the work or for inspecting the finished etching. The scribing edges must be smooth and perpendicular to the masked surface. The etch factor must be determined and designed into the scribing template.

3.3 Materials

3.3.1 Wiping Solvent - Acetone for removing primer or marking inks shall conform to Specification O-A-51C.

3.3.2 Degreasing Solvent - Trichloroethylene shall be used for liquid or vapor degreasing and shall conform to Specification O-T-634, Type II for liquid degreasing and for vapor degreasing.

3.3.3 Chem-Milled Components - All S-IC bulkhead gore segments to be etched shall have surfaces that are smooth, free of scratches, burrs, or other defects which would tend to cause uneven etching.

3.3.4 Desmutting Solution - Wyandotte 2487-B or approved alternate shall be used at ambient temperature to desmut the surface prior to chemical milling. The concentrations to be used shall be not greater than 16 ounces of Wyandotte 2487-B per gallon of solution.

3.3.5 Etchant - Turcoform No. 13 shall be mixed to a concentration of 13 ounces of etchant per gallon plus 5 ounces of Turcoform Alk-etch inhibitor per gallon. The etchant concentration shall be maintained above 11 ounces per gallon, and the inhibitor shall not exceed 20 ounces per gallon. Routine laboratory methods shall be used to determine solution strength. The concentration of the chemical milling solution shall be determined and recorded daily when gores are being chemically milled. An alternate concentration for the chemical milling solution may be used provided the procedure is submitted to the Procuring Activity for approval. Information concerning the above products may be obtained from Turco Products Inc., P. O. Box 1055, Wilmington, Delaware.

3.3.6 Maskants - Maskant material applied to the surface of a bulkhead gore shall completely protect the masked portion of the gore from the etchant during the chemical milling operation.

3.3.6.1 General - PR-785 neoprene-base maskant, or an approved alternate, shall be used for masking the S-IC gores.

3.3.6.2 Alternate Method - A form-fit neoprene rubber jacket or "boot" may be used, if available, as an alternate method of masking the back side of the gore segment. Apply suitable tape along the edge of the opening in order to evacuate air from the inside of the boot to 22 inches of Hg. Rolling is normally required to insure proper adhesion of the tape. Using neoprene cement and a brush for application, cover a narrow strip of the rubber jacket, all of the tape, and a portion of the part. Scribe part for area to be chemically milled in the usual manner.

3.3.6.3 Masking Tape - Tape employed in masking shall be preferably lead-foil tape of approximately 0.008 inch thickness and shall be equal to Scotch No. 40 electroplaters tape (Minnesota Mining and Manufacturing Company).

3.4 Chemical Milling Procedure

3.4.1 Cleaning of Bulkhead Gore Segments and Their Surface Preparation

3.4.1.1 General - To effect uniform adhesion of the organic maskant and uniform etching, bulkhead gore segments must be thoroughly clean and free of all oil, grease, ink, primer, scale, or other foreign material.

3.4.1.2 Pre-Cleaning - Bulkhead segments shall be cleaned of visible oil, grease, marking inks, forming lubricants, primers, or other contaminants by use of clean cloths or aluminum wool saturated with acetone or Type II trichloroethylene.

3.4.1.3 Degreasing - All bulkhead gore segments shall be degreased in the manner outlined in Specification MS 150.0 or equivalent.

3.4.1.4 Flash Etch - Bulkhead gore segments to be masked shall be etch cleaned by immersing in the chemical milling solution at a temperature of 195° F ($\pm 10^\circ$) for a maximum of 1 minute.

3.4.1.5 Rinse - Bulkhead gore segments shall be spray rinsed during removal from the etch tank. The gores shall then be immersed in cold flowing water and spray rinsed upon removal from the rinse tanks.

3.4.1.6 Desmut - Bulkhead gore segments shall be desmuted by immersing in a solution of the concentration and at the temperature specified in paragraph 3.3.4. Normally this operation will require less than 5 minutes.

3.4.1.7 Rinse - All bulkhead gore segments shall be rinsed in cold flowing water and thoroughly spray rinsed after removal from the rinse tank. If gores exhibit a "water-break", repeat the cleaning procedures outlined in paragraphs 3.4.1.2 through 3.4.1.7.

3.4.1.8 Drying - Bulkhead gore segments to be masked shall be allowed to evaporate to dryness in a dust-free area. After drying, gore segments shall be blown off with filtered, oil-free air to assure clean, dry surfaces for masking.

3.4.2 Masking of Bulkhead Gore Segments - Use either the type of maskant listed in paragraph 3.3.6.1 or the type listed in paragraph 3.3.6.2 of this procedure.

3.4.2.1 Thickness - The thickness of maskant desired is 5 to 8 mils for etching up to $\frac{1}{4}$ inch in depth, and 8 to 10 mils for etching over $\frac{1}{4}$ inch in depth.

3.4.2.2 Maskant Application Methods - The maskant shall be applied by a method listed in paragraphs 3.2.5.1, 3.2.5.2, 3.2.5.3, or 3.2.5.4. The maskant must be applied such that it will have sufficient strength to resist collapsing while extending beyond the already etched-out material. All edges of trimmed gore sections must be protected by lead-foil tape. The PR 785 maskant shall be allowed to air cure at ambient temperature for a minimum of 12 hours. During cold weather, it may be necessary to oven cure the maskant at 175-200° F for 2 hours.

3.4.2.3 Removal of Scribed Maskant - Care must be taken to lift the scribed maskant from areas to be etched in such a way that the surface of the gore is not scratched. Maskant that is removed shall be peeled off by working from the edges to the center of the cut-out areas. After stripping, any residual maskant must be removed by using an appropriate solvent. The remaining maskant shall be inspected for accuracy of pattern and obvious adherence to the gore segment.

3.4.2.4 Maskant Repair - Damage to the maskant, which will result in defective etched parts, must be repaired or the maskant must be stripped off completely. Damaged areas may be repaired by lead-foil tape firmly pressed in place, and edges of cut-outs may be repaired by localized spraying or brush applications of maskant.

3.4.3 Etching - The masked gore segments will be hung from a holding frame which will rest on pads at each end of the etching tank. A method of moving the part back and forth through the solution at an even rate will be employed. Lower the part into the etchant. Air agitation on both sides of the tank is necessary to obtain complete circulation of the etchant. If the alternate method of masking is used, then it will be necessary to maintain a vacuum on the neoprene boot during chemical milling. A machined 2219 alloy specimen of the same temper as the gore being etched, mounted in a spring-loaded timing device, may be attached to the holding frame and lowered into the etchant at the same time as the gore segment and signal a bell warning 5 minutes prior to final milling depth and removal from the tank. The part will be raised, desmuted, inspected, and rotated 180 degrees every 45 (± 15) minutes during the milling operation. It may be necessary to trim the maskant at this

time. After milling, all sharp chem-milled edge radii may be mechanically broken by filing, sanding, or milling to 0.010 inch minimum.

3.4.3.1 Etching Rate - Turcoform etchant No. 13 is designed to produce a cutting rate of 0.001 inch per minute; however, this will vary slightly according to concentration and temperature. Gores shall be allowed to remain in the etchant for the time required. The actual etch rate on the test specimen is described in paragraph 4.4. The amount of metal to be removed and the desired pattern will be specified on the applicable engineering drawing.

3.4.3.2 Rinse - Bulkhead gore segments shall be spray rinsed during removal from the etch tank. These gores shall then be rinsed by immersion in cold flowing water, then spray rinsed during removal from the rinse tank.

3.4.3.3 Desmutting - Smut shall be removed from the part by immersing the part in an aqueous solution of Wyandotte 2487-B, or equivalent, maintained at a concentration of up to 16 ounces per gallon and at room temperature. Immersion time will normally be less than 5 minutes.

3.4.3.4 Rinse - Gore segments shall be rinsed in cold, flowing water. Gores shall be thoroughly spray rinsed during removal from rinse tank.

3.4.4 Demasking - Stripping of maskants shall be done by hand. Gores shall be inspected for retained maskant. If any maskant has been retained, it should be removed with trichloroethylene or equivalent followed by a flash etch (20 seconds maximum), rinse, desmut, and rinse again.

4. QUALITY ASSURANCE PROVISIONS

4.1 The chemically milled bulkhead gore segments shall have a uniform smooth appearance measuring no greater than 125 microinches of surface roughness when measured by the Root Mean Square (RMS) method.

4.2 The chemical milling solution shall be maintained at a strength sufficient to perform etching at the rate of approximately 0.001 inch per minute.

4.3 When chemical milling tolerance is less than the material tolerance, the thinnest area of the material shall be used for measuring the chemical milling tolerance.

4.4 A machined test panel of the same type material and heat treat condition as the production part shall accompany each production part during etching to determine the etching rate. This panel shall be removed at the instant that it has been immersed for 1/2 of the scheduled etching time required for the production part. The panel shall be rinsed and desmutted immediately; thickness measurements shall be made.

5. NOTES

5.1 This manufacturing process has been coordinated within the Process Engineering Laboratory and is intended for use in the chemical milling of S-1C Bulkhead gores. Verification of this procedure will be established during the production of the first chemically milled bulkhead gore segments.

5.2 Any deviation from this basic procedure will require written concurrence from Process Engineering Laboratory. An emergency concurrence may be made by telephone, but it must be confirmed in writing.

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

PROCESS CONTROL AND OPERATING PROCEDURES FOR
ELECTRO CLEANING OF SPACE VEHICLE PARTS

1. SCOPE

This manufacturing process data covers the necessary precautions and procedures for process control of electrocleaning of space vehicle parts.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Handling Fixtures
- Laboratory instruments (pH meter, titrating device, etc.)
- Sampling beaker, 250 ml, minimum
- Pipette
- Erlenmeyer flask
- Graduate cylinder (100 ml)

2.2 Materials

- Proprietary cleaner solution
- pH indicator (phenolphthalein)
- Standard HCl (1.0 N HCl)

3. PROCEDURE

3.1 Solution Control and Operating Conditions

3.1.1 Agitate bath, by means of air bubbling or use of wooden paddle, for a minimum of 5 minutes, in order to obtain a composite sample.

3.1.2 Immerse (250 ml minimum) beaker and collect sample of electro-cleaner solution.

3.1.3 Pipette a 5 ml sample into a 250 ml erlenmeyer flask.

3.1.4 Add 1 ml of phenolphthalein solution to the sample.

3.1.5 Titrate in 250 ml erlenmeyer flask with standard 1.0 N HCl until the color changes from pink to colorless.

$$0.536 = \left(\frac{\text{equivalent of NaOH}}{\text{ml of sample}} \right) (0.134)$$

The sodium hydroxide in oz/gal. equals: $0.536 \times N \text{ of HCl} \times CC \text{ of HCl}$ required for sample of solution.

The figures inside the brackets represent the factor for the standard HCl solution.

3.1.6 Maintain the alkalinity between six (6) and ten (10) ounces per gallon.

4. CONTROL

4.1 Check normality of solvent against known standard once per week or as often as solutions are made-up.

4.2 Insure that beakers, pipettes and all laboratory instruments are maintained in a clean condition.

4.3 Use alternate method of electrocleaner process control as approved by Process Engineering Laboratory.

MANUFACTURING SPECIFICATION FOR CADMIUM PLATING OF ALUMINUM, COPPER, AND STEEL ALLOYS

1. SCOPE

1.1 Scope

This manufacturing specification covers the approved PE Laboratory methods and requirements for the cadmium plating of components to be used in space Launch Vehicle applications.

1.2 Applicability

The methods and requirements set forth in this specification are applicable to the following types of metals and their alloys:

- Aluminum
- Copper
- Steel

1.3 Classification

Cadmium plating shall be of the following classes and types:

Class 1	0.00050 inch thick minimum
Class 2	0.00030 inch thick minimum
Class 3	0.00020 inch thick minimum
Class 4	0.00040 inch thick minimum
Class 5	0.00010 inch thick minimum (flash coating)
Type I	Without supplementary chromate or phosphate treatment
Type II	With supplementary chromate treatment
Type III	With supplementary phosphate treatment

2. APPLICABLE DOCUMENTS

2.1 Governmental

The following documents of the issue in effect on the date of use of this specification form a part of this specification.

SPECIFICATIONS

Federal

O-A-51C	Acetone, Technical
O-A-86	Acid; Hydrochloric (Muriatic), Technical-Grade
O-A-88	Acid, Nitric; Technical - Grade
O-C-303B	Chromic Acid (Chromium Trioxide) Technical - Grade
O-H-795	Hydrofluoric Acid, Technical
O-O-670	Orthophosphoric (Phosphoric) Acid, Technical
O-S-571E	Sodium Carbonate, Anhydrous, Technical
O-S-609	Sodium Sulphate, Photographic (Anhydrous)
O-S-642	Sodium Phosphate, Tribasic, Technical, Anhydrous, Dodecahydrate, and Mono- hydrate
O-S-809a	Acid, Sulphuric; Technical - Grade
O-T634A	Trichloroethylene, Technical
P-S-631B	Sodium Hydroxide, Technical, for Cleaning Purposes
QQ-P-416a	Plating, Cadmium
QQ-A-671	Anodes, Cadmium

Military

MIL-C-6151	Cadmium Oxide
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MIL-F-14580	Ferric Chloride, Anhydrous
MIL-S-11171	Sodium Cyanide, Reagent
MIL-Z-15486A	Zinc Oxide, Technical

George C. Marshall Space Flight Center

MS 100.2	Manufacturing Specifications for the Deoxidation of Aluminum Alloys
MS 150.0	Manufacturing Specification for Vapor Degreasing Metallic Surfaces

STANDARDS

Federal

151A	Metals, Test Methods
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2.2 Other Publications

The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

E. I. duPont deNemours & Co., Inc.
Patent No. 2164924 Carbonate Remover

(Copies of the publication listed above may be procured from E. I. duPont deNemours Co., Inc., Wilmington, Delaware.)

R. O. Hull & Company, Inc.
Bulletin No. 021352 Instructions for Rohco 20-XL Addition
Agent for Perfect Cadmium Plating

(Copies of the publication listed above may be procured from the R. O. Hull & Co., Inc., 1300 Parsons Court, Rocky River 16, Ohio.)

Turco Products, Inc.
Bulletin No. 46 Turco Porokleen
Bulletin No. 84 Turco Smut-Go No. 1

(Copies of the publication listed above may be obtained from Turco Products, Inc., 24600 So. Main Street, Wilmington, California.)

3. REQUIREMENTS

3.1 General

No deviation from the requirements of this specification shall be permitted without prior written approval of the MSFC initiating activity. Approval of alternate process materials and equipment by the MSFC initiating activity implies no guarantee of acceptance of the final plated parts regardless of any process or material approval. Plating shall conform to all applicable requirements of this specification.

3.2 Equipment

3.2.1 Cleaning and plating tanks

Tanks of a suitable size, constructed of heavy-gauge, hot-rolled steel sheets with joints welded inside and out shall be used for containing basic cleaning and plating solutions. Sulphuric acid solutions shall be contained in lead-lined steel tanks. Nitric acid solutions shall be contained in tanks lined with Type 347 stainless steel. Hydrofluoric acid solutions shall be contained in steel tanks lined with a suitable plastic, such as Koroseal or Teflon, or with carbon brick; preferably a combination of both.

3.2.2 Bars, hangars, racks, and baskets

Tanks shall be equipped with the proper types of bars, hangars, racks, and baskets to assure an efficient cleaning and plating operation.

3.2.2.1 Aluminum alloys

Aluminum racks shall be used in the plating of aluminum alloys. Aluminum racks shall have spines fabricated from 1100 alloy and contacts fabricated from 2024 alloy.

3.2.2.2 Copper and steel alloys

Copper or steel racks shall be used in the plating of copper and steel alloys, respectively.

3.3 Materials

3.3.1 Phosphoric acid

Phosphoric acid shall be Class B of Specification O-P-313 (1).

3.3.2 Sulphuric acid

Sulphuric acid shall conform to Specification O-S-809a, Type I, Class 1.

3.3.3 Carbonate remover

The carbonate remover shall be a commercial product equivalent to the remover furnished by E. I. duPont Co.

3.3.4 Plating brightener

This material shall be used in the plating bath to assist in obtaining a bright cadmium finish and shall be equivalent to Rohco 20 XL, as supplied by the R. O. Hull Company.

3.3.5 Masking tape

Masking tape shall be Minnesota Mining and Manufacturing Company No. 470 Electroplaters Tape, or equal.

3.3.6 Etchant

A product used for etching the surface of aluminum and removing any oxide film shall be available. It shall be equivalent to Turco 2897.

3.4 General Procedure

3.4.1 Preparation of Solutions

3.4.1.1 Prefilling tanks

Tanks shall be filled approximately 1/2 full with tap water.

3.4.1.2 Chemical additions

The required chemicals shall be added and the tanks stirred thoroughly.

NOTE: Do not use air agitation in the plating tank.

3.4.1.3 Filling tanks

The tanks shall be filled to the desired level with tap water and thoroughly mixed.

3.4.1.4 Tank temperature

All tanks shall be heated to and operated at the specified temperature.

3.4.1.5 Anodes

Cadmium anodes shall be suspended in the solution from the tank bus bars in basket-type containers. Approximately 2 pounds of cadmium anodes per gallon of solution are required for general plating at full capacity. This may be varied for plating special surfaces.

3.4.1.6 Carbonate removal

Excess carbonate may be removed with a suitable carbonate remover or by chilling and filtering. The carbonates shall be between the limits of 2 to 8 oz/gal.

3.4.1.7 Brighteners

Brightening agents shall be added as necessary or as required by the type plating being applied.

3.4.2 Typical cadmium plating solution analysis

Cadmium oxide - 3.0 to 5.2 oz/gal.

Sodium cyanide - 11.5 to 17.5 oz/gal.

Brightener (general) - as required.

Sodium carbonate - 2.0 to 8.0 oz/gal.

3.4.3 Fabrication of components

Unless otherwise specified, cadmium plating shall be applied only after all machining, brazing, welding, forming, and perforating of the component have been completed.

3.4.4 Cleaning of components

3.4.4.1 General

All metal surfaces shall be thoroughly cleaned prior to the application of the cadmium plate. Meticulous cleaning and surface preparation of the components cannot be overemphasized since this factor is of prime importance in obtaining a satisfactory plate cover.

3.4.4.2 Cleaning and surface preparation of steels containing 0.35 percent carbon or more and case-hardened steels containing less than 0.35 percent carbon

A. Precleaning - Using clean cloths or steel wool saturated with acetone or trichloroethylene, remove all inked lettering, wax pencil or crayon markings, oil, grease, forming lubricants, caked-on dirt, drawing compounds, burnt-in oil, heavy scale, and emery and steel particles.

B. Degrease - Degrease all material by immersion in clean, hot trichloroethylene, by spray-washing, by emulsion-cleaning, or by vapor degreasing.

C. Alkaline clean - Parts that are to be barrel-plated shall be cleaned by soaking or tumbling in an aqueous solution containing 4 to 8 ounces of Turco Porokleen, or equal, per gallon of solution at a temperature of 180 to 210 degrees F for 5 to 10 minutes. All other parts shall be cleaned in an electrolytic anodic alkaline cleaner bath containing 12 to 16 ounces of Turco Porokleen, or equal, per gallon of solution at a temperature of 200 to 210° F and at a minimum current density of 50 amperes per square foot for a period of time not to exceed 2 minutes.

D. Rinse - Upon removal from the alkaline anodic cleaning bath, parts shall be immediately rinsed by immersion in clean tap water at ambient temperature for 10 to 15 seconds. In addition, parts shall be subjected to a tap water spray as they enter and leave the rinsing tank.

E. Stress relieve - All ferrous parts having a hardness greater than Rockwell C-40 shall be stress relieved by heating to a temperature of 400° F and maintaining at that temperature for at least 1/2 hour.

F. Mechanical cleaning - Small parts shall be mechanically cleaned by tumbling with or without an abrasive. Parts too large for tumbling shall be mechanically cleaned by sand, grit, or vapor blasting. For parts where the surface smoothness or dimensions are of critical importance, grinding may be used for mechanical cleaning.

G. Stress relieve - After mechanical cleaning, parts having a hardness greater than Rockwell C-40 shall again be stress relieved in the manner specified in paragraph 3.4.4.2.E.

H. Electrolytic anodic cleaning - All parts too large for barrel cleaning shall be cleaned in an electrolytic anodic alkaline cleaner bath containing 8 to 16 ounces of Turco Porokleen, or equal, per gallon of solution at a temperature of 190 to 210° F and at a minimum current density of 50 amperes per square foot for a period of time not to exceed 2 minutes.

I. Rinse - Upon removal from the electrolytic anodic cleaner, parts shall be immediately rinsed by immersion in clean tap water at a temperature of 120° F ($\pm 5^\circ$) for 2 to 5 minutes. Following this, parts shall be additionally rinsed by spray rinsing with fresh tap water at ambient temperature for 1 to 2 minutes.

J. Oxide removal - Last traces of surface oxides shall be removed by immersion of parts in an aqueous solution containing 25 percent by volume of hydrochloric acid (sp. gr. 1.158) at ambient temperature for 15 to 30 seconds.

K. Rinse - All parts shall be rinsed in the manner specified in paragraph 3.4.4.2.D.

L. Smut removal - Smut, resulting from the oxide removal step, shall be removed from all parts by a 1/2 to 1 minute anodic treatment at 15 to 20 amperes per square foot at 70° F in an aqueous solution containing 6 ounces of sodium cyanide per gallon of solution.

M. Rinse - After smut removal, all parts shall be rinsed again in the manner specified in paragraph 3.4.4.2.D.

N. Anodic acid etching - All parts shall be anodically acid etched in an aqueous solution containing 30 to 32 percent by volume of sulphuric acid (66° Baume) and 1.0 to 1.1 pounds of sodium sulphate (anhydrous) per gallon of solution at a temperature of 70 °F ($\pm 5^\circ$). Anodic treatment shall be at a current density of 150 (± 50) amperes per square foot for a period of time not to exceed 1 minute.

O. Rinse - After anodic acid etching, all parts shall be rinsed again in the manner specified in paragraph 3.4.4.2.D.

P. Final oxide removal - Light airformed oxides shall be removed by dipping all parts into an aqueous solution containing 3 ounces of sodium cyanide per gallon of solution at ambient temperature for 15 to 30 seconds.

NOTE: Do not allow parts to dry or to be exposed to air any more than absolutely necessary between final oxide removal and plating.

Q. Plating - Cleaned high-carbon steel parts shall be plated in the manner specified in paragraph 3.4.6.

3.4.4.3 Cleaning and surface preparation of steels containing less than 0.35 percent carbon

A. Stress relief - Ferrous parts having a hardness greater than Rockwell C-40 shall be stress relieved by heating to a temperature of 400° F and maintaining at temperature for at least 1/2 hour.

B. Precleaning - Steel parts shall be precleaned in the manner specified in paragraph 3.4.4.2.A, or by spray cleaning in a power washer using an alkaline or emulsion-type cleaner at a nozzle pressure of 30 to 60 psi.

C. Degrease - Vapor degrease all parts, having recesses or overlapping joints, by exposure to trichloroethylene vapors at 188° F ($\pm 3^\circ$) until vapors cease to condense on the part.

D. Alkaline clean - Parts shall be anodically cleaned in an alkaline bath in the manner specified in paragraph 3.4.4.2.C.

E. Rinse - Upon removal from the alkaline anodic cleaning bath, parts shall be immediately rinsed by immersion in clean tap water at ambient temperature for 10 to 15 seconds. In addition, parts shall be subjected to a tap water spray as they enter and leave the rinsing tank.

F. Oxide, scale, and rust removal - Oxide, scale, and rust shall be removed from all parts by immersing them in an aqueous solution containing 25 to 85 percent by volume of hydrochloric acid (sp. gr. 1.158) at ambient temperature for the length of time necessary to remove all oxide, scale, and rust.

G. Rinse - Upon removal from the acid pickling bath, parts shall be immediately rinsed in the manner specified in paragraph 3.4.4.3.E, but shall be rinsed in a separate rinsing tank.

H. Anodically clean - Subject all parts to an anodic cleaning cycle in the manner specified in paragraph 3.4.4.2.C, but in a separate cleaning tank.

I. Rinse - Rinse parts again in the manner specified in paragraph 3.4.4.3.E, but in a separate rinse tank.

J. Surface activation - Activate steel surfaces for plating by immersion of all parts in an aqueous solution containing 4 to 10 percent by volume of sulphuric acid (66° Baume) at ambient temperature for 5 to 15 seconds.

K. Rinse - Rinse parts again in the manner specified in paragraph 3.4.4.3.E, but in a separate rinse tank.

NOTE: Do not allow parts to dry or to be exposed to air any more than absolutely necessary between the final rinse and plating. If necessary, temporarily store the parts in an aqueous solution containing 2 to 4 ounces of sodium cyanide and 2 to 4 ounces of sodium hydroxide per gallon of solution at ambient temperature.

L. Plate - Cleaned low-carbon steel parts shall be plated in the manner specified in paragraph 3.4.6.

3.4.4.4 Cleaning and surface preparation of stainless steels

A. Stress relief - Stainless steel parts having a hardness greater than Rockwell C-40 shall be stress relieved by heating to a temperature 300 to 700° F for 1 to 2 hours in an air furnace.

B. Scale breakup - Mill, heat-treating, or processing scale and adherent oxides shall be broken up by dry or vapor blasting, shot peening, or tumbling preparatory to complete removal by pickling.

C. Scale removal — Final traces of mill, heat-treating, or processing scale and adherent oxides shall be completely removed by two-step acid pickling or by molten salt treatment.

(1) Two-step acid pickling

- Immerse parts in an aqueous solution containing 8 to 11 percent by volume of sulphuric acid (sp.gr. 1.83) at 150 to 160°F for 10 to 45 minutes or in an aqueous solution containing 10 to 15 percent by volume of hydrochloric acid (sp.gr. 1.158) at 120 to 140°F for 30 to 90 minutes.

- Rinse parts thoroughly by immersion and spray rinsing with ambient temperature tap water.

- Immerse parts in an aqueous solution containing 6 to 10 percent by volume of nitric acid (sp. gr. 1.38) and 1-1/2 percent by volume of hydrofluoric acid (sp.gr.1.20) at 70°F ($\pm 5^\circ$) for 10 to 30 minutes, or until all smut has been removed.

- Rinse parts thoroughly by immersion and spray rinsing with ambient temperature tap water.

(2) Molten salt treatment

- Immerse parts in a molten sodium hydroxide bath containing 1-1/2 to 2 percent by weight of sodium hydride at 750° F for 1 to 15 minutes followed by quenching in room temperature tap water.

- Dip for 15 to 60 seconds in an aqueous solution containing 10 percent by volume of sulphuric acid (sp. gr. 1.83) at room temperature.

- Rinse parts thoroughly by immersion and spray rinsing with ambient temperature tap water.

- Brighten the stainless steel surface by immersion in an aqueous solution containing 10 to 20 percent by volume of nitric acid (sp. gr. 1.38) at 160 to 180°F for 5 to 10 minutes, or in a solution containing 10 to 20 percent by volume of nitric acid (sp. gr. 1.38) and 1-1/2 to 2 percent by volume of hydrofluoric acid (sp.gr. 1.20) at 160 to 180°F for 5 to 10 minutes.

- Rinse parts thoroughly by immersion and spray rinsing with ambient temperature tap water.

D. Degrease - Vapor degrease all parts by exposure to trichloroethylene vapors at 188°F ($\pm 3^\circ$) until vapors cease to condense on the metal surfaces.

E. Removal of metal lubricants - Metal lubricants such as copper, lead, or cadmium applied to stainless steel for forming purposes shall be removed by immersion of parts in an aqueous solution containing 15 to 25 percent by volume of nitric acid (sp. gr. 1.38) at 120 to 140°F for 5 to 15 minutes.

F. Alkaline clean - Parts shall be anodically cleaned in an alkaline bath in the manner specified in paragraph 3.4.4.2.C.

G. Rinse - Upon removal from the alkaline anodic cleaning bath, parts shall be immediately rinsed by immersion in clean tap water at ambient temperature for 10 to 15 seconds. In addition, parts shall be subjected to a tap-water spray as they enter and leave the rinse tank.

H. Surface activation - Surfaces of all parts shall be activated prior to plating by subjecting the parts to a cathodic current density of 5 to 10 amperes per square foot for 1 to 5 minutes at 70°F ($\pm 5^\circ$) in an aqueous solution containing 5 to 50 percent by volume of sulphuric acid (sp. gr. 1.83).

I. Rinse - After surface activation parts shall be immediately rinsed in the manner specified in paragraph 3.4.4.4.G.

NOTE: Do not allow parts to dry or to be exposed to air any more than absolutely necessary between the final water rinse and plating. If necessary, temporarily store the parts in an aqueous solution containing 2 to 4 ounces of sodium cyanide and 2 to 4 ounces of sodium hydroxide per gallon of solution at room temperature.

J. Plate - Cleaned and activated stainless steel parts shall be plated in the manner specified in paragraph 3.4.6.

3.4.4.5 Copper cleaning and preparation (oxide coated and oil contaminated)

A. Degrease - Degrease copper material and components by immersion in liquid trichloroethylene at 185 to 195° F for 2 to 5 minutes followed by rinsing in cooled, condensed trichloroethylene liquid at 70 to 100° F for 1 to 2 minutes followed by vapor degreasing in the manner specified in Specification MS 150.0.

B. Alkaline clean - Alkaline clean copper material or components by immersion in an aqueous solution containing 4 to 6 ounces of Turco Aviation, or equal, per gallon of solution at 175 to 185° F for 5 to 15 minutes.

C. Rinse - Rinse copper components by immersion in tap water at 140 to 160° F for 1 to 2 minutes. Spray rinse the parts with clean tap water at 70 to 140° F as they are being removed from the rinse tank.

D. Electroclean - Cathodically electroclean copper components by immersion in an aqueous solution containing 6 to 8 ounces of Turco Porokleen, or equal, per gallon of solution at 160 to 180° F for 2 to 3 minutes.

E. Rinse - Rinse copper components in the manner specified in paragraph 3.4.4.5.3.

F. Oxide removal - Remove any oxides from the surfaces of copper components by immersion in an aqueous solution containing 5 to 10 percent by volume of sulphuric acid (sp. gr. 1.84) and 4 to 8 ounces of sodium dichromate per gallon of solution at 70 to 175° F for 1 to 15 minutes.

G. Rinse - Rinse copper components in the manner specified in paragraph 3.4.4.5.C.

H. Bright dip - Brighten copper components by immersion in a solution containing 5 to 10 percent by volume of water, 60 to 75 percent by volume of sulphuric acid (sp. gr. 1.84), 20 to 35 percent by volume of nitric acid (sp. gr. 1.42), and 1/8 fluid ounce of hydrochloric acid (sp. gr. 1.158) per gallon of solution at a temperature of 70 to 80° F for 5 to 15 seconds.

I. Rinse - Rinse copper components by double immersion in two separate tap-water rinse tanks at 70 to 80° F or by thoroughly spray rinsing with tap water at 70 to 80° F.

J. Cyanide dip - Immerse copper components in an aqueous solution containing 4 to 6 percent by weight of sodium cyanide plus 2 to 4 ounces of sodium hydroxide per gallon of solution at 70 to 80° F for 15 to 30 seconds.

NOTE: Do not allow the copper components to dry or to be exposed to air any longer than absolutely necessary between the time the component is removed from the cyanide dip and placed in the cyanide plating bath.

K. Plate - Cadmium plate clean copper components in the manner specified in paragraph 3.4.6.

3.4.4.6 Aluminum cleaning and preparation

A. Degrease - Aluminum components shall be degreased in the manner specified in paragraphs 3.3.1 through 3.3.7 of Specifications MS 150.0.

B. Alkaline clean - Alkaline clean aluminum components in the manner specified in paragraph 3.4.4 of Specification MS 100.2A.

C. Spray rinse - Spray rinse components with ambient temperature tap water while slowly raising and removing the part from the 4215 alkaline cleaner tank. If water breaks appear on material surfaces after rinsing, repeat paragraphs 3.4.4.6.A through 3.4.4.6.C until water breaks no longer appear.

D. Deoxidize and condition - Aluminum components shall be deoxidized in the manner specified in paragraph 3.4.7 of Specification MS 100.2A and conditioned in the applicable manner specified below.

(1) A13, 319, 356, and 380 casting alloys - Dip at ambient temperature for 3 to 5 seconds in an aqueous solution containing 3 percent by volume of nitric acid (sp. gr. 1.42) plus 1 percent by volume of hydrofluoric acid (48 percent).

(2) All wrought alloys and cast alloys other than those specified in 3.4.4.6.D(1) - Immerse in an aqueous solution containing 15 percent by volume of sulphuric acid (66°Baume) for a period of 2 to 5 minutes at a temperature of 175 to 180 °F.

(3) Cast alloys, wrought alloys not containing magnesium, and unidentified alloys - Immerse in an aqueous solution containing 70 ounces of commercial sodium hydroxide (76 percent Na_2O), 13 ounces of zinc oxide (technical grade), 0.13 ounce of ferric chloride (anhydrous), and 1.3 ounces of Rochelle salts per gallon of solution at a temperature of 70°F ($\pm 10^\circ$) for 30 to 60 seconds. Rinse the component by immersion in ambient temperature tap water for 1 to 2 minutes. Remove the zinc layer by dipping the component into an aqueous solution containing 50 percent by volume of nitric acid (sp. gr. 1.37) at ambient temperature for 1 to 2 minutes.

E. Rinse - Thoroughly rinse all components by immersion in clean tap water at ambient temperature for 1 to 2 minutes.

F. Zinc immersion coating - Immerse all components in an aqueous solution containing 70 ounces of commercial sodium hydroxide (76 percent Na_2O), 13 ounces of zinc oxide (technical grade), 0.13 ounce of ferric chloride (anhydrous), and 1.3 ounces of Rochelle salts per gallon of solution at a temperature of 70°F ($\pm 10^\circ$) for 30 to 60 seconds.

G. Rinse - Double rinse all components by means of two combination "dip and spray" rinses using tap water at ambient temperature until all traces of the zincate solution have been removed.

NOTE: Do not allow parts to dry between final rinsing (paragraph 3.4.4.6.G) and plating.

H. Plate - All aluminum components that have been cleaned and conditioned as specified in paragraphs 3.4.4.6.A through 3.4.4.6.G shall be connected to the bus bar and the plating current applied before immersion in the plating bath and plating as specified in paragraph 3.4.6.

3.4.5 Masking

Components requiring only partial or special area plating shall be masked with Minnesota Mining and Manufacturing Company No. 470 Electroplaters Tape, or equal, over those areas which do not require plating.

3.4.6 Plating

3.4.6.1 General

With all base metals except aluminum and stainless steel, cadmium plating shall be deposited directly on the basic metal without a preliminary

plating of any other metal, such as nickel. Aluminum shall have a preliminary zinc immersion coating prior to cadmium plating. Components made wholly or in part of corrosion resistant steel may have a preliminary plating of nickel prior to cadmium plating.

3.4.6.2 Appearance

Cadmium plating shall be a smooth, adherent, fine-grained homogeneous deposit free from blisters, nodules, porosity, pits, or other harmful defects. Superficial stains or slight discolorations resulting from rinsing or racking shall not be cause for rejection. Plating accomplished without the use of brighteners shall have a dull gray matte finish.

3.4.6.3 Cadmium electroplate

Components that have been cleaned and prepared for plating in conformance with paragraphs 3.4.4 and 3.4.5 shall be plated to the desired thickness by applying enough voltage to obtain the desired amperage. They shall be immersed in the plating bath for the specified time to obtain the required thicknesses.

3.4.6.4 Thickness of plating

Plating thickness shall in all cases conform to the requirements of the applicable engineering Drawing or engineering order.

A. Minimum thickness when not specified on drawing - The minimum thickness of cadmium plate shall be 0.0003 inch except on:

- Externally threaded portions of articles where the tolerances of the thread preclude deposition of 0.0003 inch but, unless otherwise specified, not less than 0.0002 inch thick.
- Parts whose dimensional tolerances will not permit a thickness of 0.0003 inch but shall not be less than a maximum compatible with the dimensional tolerances.
- Recesses, holes, internal threads, and other areas where controlled deposits cannot be obtained under normal plating conditions. These areas shall be protected by coatings such as phosphate, chromadize, conversion, or others as may be specified by an engineering order.

- Washers, studs, bolts, nuts, and other articles with major portions externally threaded, but, unless otherwise specified, not less than 0.0002 inch thick.

B. Maximum thickness when not specified on drawing - Where a dimensional tolerance is not specified, the maximum thickness shall be that thickness which does not interfere with the function of the part; however, no plate shall be applied over 0.0005 inch thick except on approval by the initiating activity.

3.4.6.5 Rinse

Parts shall be rinsed thoroughly in water as soon as they are removed from the electroplating bath. Aluminum parts shall be finally rinsed with tap water heated to 120 to 140 °F. After rinsing in water, plated parts shall be brightened by dipping into an agitated aqueous solution containing 3 to 5 percent by weight of chromic acid at room temperature.

3.4.7 Plate removal

3.4.7.1 Steel and copper

If it is found necessary to remove cadmium plate, it shall be stripped from the base metal by immersion, with proper agitation, for the needed length of time in a solution containing 1 pound of ammonium nitrate per gallon of solution.

3.4.7.2 Aluminum

If stripping of cadmium plating from aluminum is required, stripping shall be accomplished by reversing the current (make the plated part the anode) in a regular cyanide plating bath, or in an aqueous solution containing 60 percent by volume of sulphuric acid (sp. gr. 1.83) at room temperature, or in an aqueous solution containing 75 percent by volume of phosphoric acid (Class B) at room temperature until all cadmium plating is removed.

3.5 Hydrogen Embrittlement Removal

3.5.1 Cadmium plated steel

Within 1 hour after the completion of the cadmium plating operation, steel components shall be placed in a furnace and heated in the manner specified below.

3.5.1.1 Parts having a hardness of Rockwell C-35 to C-40

Parts having a hardness of Rockwell C-35 to C-40 shall be heated to 375° F ($\pm 25^\circ$) and maintained at temperature for a minimum of 4 hours.

3.5.1.2 Parts having a hardness greater than Rockwell C-40

Parts having hardnesses greater than Rockwell C-40 shall be heated to 375° F ($\pm 25^\circ$) and maintained at temperature for a minimum of 24 hours.

3.6 Surface Treatments After Plating

3.6.1 Chromate treatment (Type II cadmium plate)

When a Type II cadmium plate is specified on the applicable engineering drawing, the required chromate conversion coating shall be produced in the manner specified below.

3.6.1.1 Cleaning

The cadmium plated surfaces to be chromated shall be cleaned by vapor degreasing at 188° F ($\pm 3^\circ$) or by emulsion cleaning using Turco Borzin, or equal, at 70 to 120° F.

3.6.1.2 Rinse

Components cleaned by vapor degreasing do not require a water rinse. Components cleaned by emulsion cleaning shall be rinsed by spraying with high pressure tap water at 70 (± 5) psi and at a flowrate of 150 gallons per minute until all traces of the emulsion cleaner have been removed. After spraying, components shall be additionally rinsed by immersion in clean, running tap water at room temperature for 2 to 5 minutes.

3.6.1.3 Chromating

Immerse components into, or spray onto components, Iridite No. 1, or equal, or an aqueous solution containing from 1 to 70 grams per liter of chromic acid at a temperature of 60 to 120°F for a period of time from 5 seconds to 10 minutes and at a solution pH from below 0 to 3.5.

3.6.1.4 Water rinse

After chromating, components shall be immediately rinsed by immersion in fresh running tap water at 70 to 150° F until all traces of excess solution from the chromating bath have been removed.

3.6.1.5 Drying

Component surfaces shall be thoroughly dried statically in air at 70° F ($\pm 5^\circ$) for a minimum of 24 hours or by exposing to air heated to a maximum of 150° F for a sufficient length of time to thoroughly dry all surfaces.

3.6.2 Phosphate treatment (Type III cadmium plate)

When a Type III cadmium plate is specified on the applicable engineering drawing, the required phosphate conversion coating shall be produced in the manner specified below.

3.6.2.1 Cleaning

Cadmium plated surfaces to be phosphated shall be cleaned in the manner specified in paragraph 3.6.1.1.

3.6.2.2 Water rinse

Cleaned components shall be rinsed in the manner specified in paragraph 3.6.1.2.

3.6.2.3 Phosphating

Immerse components into, or spray onto components, an aqueous solution containing from 0.6 to 1.0 percent by volume of phosphoric acid (Class B), plus 5 to 10 percent by weight of zinc phosphate, and 1 to 2 percent by weight of nickel nitrate at a temperature of 125 to 180° F for 1 to 3 minutes.

3.6.2.4 Water rinse

While still wet from the phosphating bath, components shall be rinsed in the manner specified in paragraph 3.6.1.4.

3.6.2.5 Chromic acid rinse

While still wet from the final water rinse, components shall be immersed for a minimum of 1 minute in a 140 to 210°F aqueous solution maintained at a pH of 2.0 to 4.0 by the addition of chromic acid or mixtures of chromic and phosphoric acids.

3.6.2.6 Drying

Component surfaces shall be thoroughly dried by exposure to either still or moving air at temperatures up to but not exceeding 250°F.

3.7 Adhesion of Plating

Adhesion of all cadmium plating shall be such that when examined at a magnification of 4 diameters it does not show separation from the basis metal at the interface when tested as specified in paragraph 4.3.2. The interface between the cadmium and the basis metal is defined as the surface of the basis metal before plating. The formation of cracks in the cadmium plate, caused by the rupture of the basis metal, which do not result in flaking, peeling, or blistering of the plate shall not be considered as lack of adhesion.

3.8 Thickness of Plating

Unless otherwise specified, the thickness of cadmium plating shall be as specified in paragraph 1.3 on all visible surfaces which can be touched by a ball 0.75 inch in diameter.

3.9 Corrosion Resistance

Type II cadmium plating of all classes shall show no white corrosion products after continuous exposure to salt spray. The appearance of white corrosion products, visible at a distance of 20 inches to a person with 20/20 vision and without the use of optical magnification, at accidental scratches through the chromate film or at unscratched areas of the chromate film shall be cause for rejection, except that white corrosion products at edges shall not be considered as failure of the chromate coating.

3.10 Paint Adhesion

Type III cadmium plating of all classes shall afford a good base for

paint. Paint coatings applied to Type III cadmium plate shall show no blistering or loss of adhesion to the phosphate coating after soaking in distilled water for 24 hours.

4. MANUFACTURING CONTROL PROVISIONS

4.1 Test Specimens

Plated articles not readily adaptable to a test specified herein, shall be tested using separate specimens plated with the articles they represent. Prior to plating, separate specimens shall be of the same basis metal, of the same form (casting, wrought, or forged), and of the same surface finish as the articles they represent, except that hot rolled specimens may be used to represent cast or forged articles. Separate specimens shall be approximately 1 inch wide, 4 inches long, and 0.040 inch thick for adhesion tests; but shall be at least 4 inches wide, 6 inches long, and approximately 0.040 inch thick for all other tests. Separate specimens shall be introduced into a group of parts at regular intervals prior to the cleaning operations preliminary to plating and shall not be removed therefrom until after completion of the plating process. Conditions affecting plating of the specimens, including the spacing and positioning with respect to anodes and to other objects being plated, shall correspond as nearly as possible to those affecting the significant surfaces of the articles represented.

4.2 Sampling

4.2.1 Lot

For the purposes of this specification, a lot is defined as a group of plated articles of the same size and the same shape having the same type and class of cadmium plating, and submitted for inspection at the same time.

4.2.2 Sampling for visual inspection and for non-destructive tests of plating thickness

A sample shall be drawn from each inspection lot by taking at random from the lot the percentage of articles specified in Table 4-5. These articles shall then be subjected to both the visual inspection and the plating thickness determination outlined in paragraphs 4.2.2.1 and 4.2.2.2, respectively.

4.2.2.1 Visual examination

All the sample articles selected in paragraph 4.2.2 shall be visually

TABLE 4-5. SAMPLING FOR NON-DESTRUCTIVE TESTS

ITEMS IN INSPECTION LOT (NUMBER)	ITEMS IN SAMPLE (PERCENT OF INSPECTION LOT)		ACCEPTANCE NUMBER (MAXIMUM ALLOWABLE NUMBER OF ITEMS NONCONFORMING)
	Visual, 4.2.2.1	Thickness, 4.2.2.2	
1 to 6	100	100	0
7 to 15	100	100	0
16 to 40	100	100	0
41 to 110	100	60	0
111 to 300	60	40	1
301 to 500	30	20	1
501 and over	20	15	2

examined to assure conformance to the requirements of paragraph 3.4.6.2. If the number of non-conforming articles exceeds the acceptance number for that size sample (see Table 4-5), the lot represented by that sample shall be rejected.

4.2.2.2 Thickness determination

All the sample articles selected in paragraph 4.2.2 shall have their plating thickness measured by a non-destructive method at several locations on each article where the plating is expected to be of minimum thickness. An article shall be considered to be nonconforming if any measurement fails to meet the specified minimum thickness. If the number of nonconforming articles in any sample exceeds the acceptance number for that size sample (see Table 4-5), the lot represented by that sample shall be rejected.

4.2.3 Sampling for destructive tests of plating thickness, adhesion, salt spray, and water resistance

A random sample of the size specified in Table 4-5, but composed of different articles than those selected for non-destructive tests in paragraph 4.2.2, shall be selected from each inspection lot and eight separately plated test specimens shall be prepared to represent each inspection lot.

4.2.3.1 Thickness tests

Each article of the sample selected in 4.2.3 shall be tested in several locations at which the plating would be expected to be a minimum. If the plating thickness at any location on any article is less than the specified thickness, the lot represented by that article shall be rejected.

4.2.3.2 Salt spray resistance

For Type II cadmium plating, four of the separately plated test specimens prepared in paragraph 4.2.3 shall be tested in lieu of plated articles which comprise the inspection lot. The failure of any test specimen in less than the minimum times specified in Table 4-7 for the class of plating being tested shall result in the rejection of the lot represented by that test specimen.

4.2.3.3 Adhesion

All articles used for plating thickness tests, if of suitable size and form, shall be used as specimens for the adhesion tests. If the articles used

for plating thickness tests are not of suitable size and form, an equal number of additional articles shall be selected or separately plated test specimens used. Failure of any test specimen shall result in the rejection of the lot represented by that test specimen.

4.2.3.4 Water resistance

A random sample of Type III cadmium plated articles of the sample size specified in Table 4-6, or an equal number of 4- by 8-inch test panels, shall be subjected to the water resistance test described in paragraph 4.3.4. Failure of any of the test articles or panels shall result in the rejection of the lot represented by that test article or panel.

4.2.3.5 Hydrogen embrittlement relief

A random sample of the size specified in Table 4-6 shall be selected from each lot of plated articles and tested for hydrogen embrittlement relief in the manner specified in paragraph 4.3.5. Failure of any of the articles tested shall result in the rejection of the lot represented by that article.

4.3 Test Methods

4.3.1 Thickness

Thickness determinations shall be made by the drop test, by the microscopic method, or by an instrument or electrochemical method approved by the MSFC Initiating Activity. For referee tests, the microscopic method shall be used. Measurement of minimum plating thickness on bolts shall be made on the shank adjacent to the thread, or on the head if the shank is completely threaded. Thickness measurements of Type II and Type III cadmium plating shall be made after the application of Type II and Type III supplementary coatings and subsequent removal of these coatings. The chromate film may be removed from Type II plating by using a paste of levigated alumina rubbed on with the finger. The phosphate coating may be removed from Type III plating by scrubbing with a rubber policeman in a 10 percent solution of NaOH for 10 to 15 minutes.

4.3.1.1 Drop test

A. Equipment - The equipment for the drop test shall consist of a 250-milliliter container having an outlet tube equipped with one stopcock and having the discharge end of the outlet tube drawn to a small diameter so as to deliver drops of approximately 0.05 milliliter each when the stopcock is fully open.

TABLE 4-6. SAMPLING FOR DESTRUCTIVE TESTS

ITEMS IN INSPECTION LOT (NUMBER)	ITEMS IN SAMPLE (NUMBER)	ACCEPTANCE NUMBER (MAXIMUM ALLOWABLE NUMBER OF ITEMS NONCONFORMING)
1	0	0
2 to 5	1	0
6 to 25	3	0
26 to 100	4	0
101 to 300	6	0
301 to 5000	10	0
5001 and over	15	0

TABLE 4-7. PLATING PERFORMANCE AND SALT SPRAY
CORROSION RESISTANCE TIME MINIMUMS

MINIMUM TIME FOR TEST SOLUTION TO PERFORATE PLATING (SECONDS)	CLASS OF PLATING	MINIMUM TIME UNTIL APPEARANCE OF WHITE CORROSION PRODUCTS (HOURS)
38	Class 1	96
22	Class 2	96
16	Class 3	96
30	Class 4	96
8	Class 5	96

TABLE 4-8. PLATING TIMES AND ELECTRICAL REQUIREMENTS

DESIRED THICKNESS (INCH)	TIME (MINUTES)	AMPERES PER SQUARE FOOT	APPROXIMATE VOLTS
.0001"	3	20	1.3
.0002"	9	15	1.0
.0002"	6 1/2	20	1.3
.0002"	5 1/2	25	1.5
.0003"	13 1/2	15	1.0
.0003"	10	20	1.3
.0003"	8 1/2	25	1.5
.0004"	18	15	1.0
.0004"	13 1/2	20	1.3
.0004"	11	25	1.5
.0005"	20	15	1.3

B. Test Solution - The test liquid for the drop test shall be an aqueous solution containing 200 grams of chromic acid and 27 milliliters of sulphuric acid (sp. gr. 1.84) per liter of solution.

C. Procedure - The plated article or test specimen and the test solution shall be brought to a temperature of 75° F ($\pm 5^\circ$). The plated surface to be tested shall be cleaned until free from water break by rinsing with an organic solvent, rubbing lightly with pure magnesium oxide paste, rinsing, drying, and bringing back to 75 degrees F (± 5 degrees). The flow rate of test solution from the container outlet tube shall be adjusted to 100 (± 5) drops per minute. The cleaned article shall be supported so that the test surface is at an angle of 45 (± 1) degrees from the horizontal then moved to a position $7/8$ ($\pm 1/8$) inch below the discharge end of the outlet tube. The elapsed time, in seconds, from the time the first drop strikes the surface until the basis metal first appears shall be determined with a stop watch. The spent solution shall not be used for subsequent thickness tests. Adequacy of plating thickness shall be determined by reference to Table 4-7 for the several classes of plating.

4.3.1.2 Microscopic test

A. Specimens - Specimens for microscopic measurements shall consist of cross-sections of plated articles, or test specimens, suitably mounted, ground, polished, and etched to differentiate the cadmium plating from the basis metal.

B. Metallurgical microscope - The plating thickness of prepared specimens may be measured by means of a metallurgical microscope equipped with a filar micrometer ocular which has been calibrated against a standard scale to an accuracy of ± 2 percent.

C. Metallographic microscope - The plating thickness of prepared specimens may be measured by means of a metallographic microscope by projecting the image of the specimen on the ground-glass focusing plane of the camera at a known magnification. The actual plating thickness can then be easily determined by measuring the plating thickness of the projected image in inches, and dividing this measured value by the known image magnification.

4.3.2 Adhesion

4.3.2.1 Scrape method

Adhesion may be determined by scraping the surface of the plated article, or test specimen, to expose the basis metal. The junction of the plating and the exposed basis metal shall then be examined at four diameters magnification. Any evidence of flaking, blistering, or separation between the plating and the basic metal shall be interpreted as unsatisfactory adhesion.

4.3.2.2 Bend method

Adhesion may be determined by clamping the plated article, or test specimen, in a suitable holding device and rapidly bending at room temperature through an angle of 180 degrees, around a diameter equal to the thickness of the specimen, until plating rupture occurs. If the edge of the ruptured plating can be peeled back or if separation between the plating and the basic metal can be seen at the point of rupture when examined at four diameters magnification, adhesion shall be interpreted as unsatisfactory.

4.3.3 Salt spray resistance

Salt-spray resistance tests shall be made in the manner specified in Method 812 of Federal Test Method Standard Number 151a. Type II cadmium plating shall be aged in air at room temperature for a minimum of 24 hours after chromating before it is subjected to salt spray.

4.3.4 Water resistance

Plated articles, or test specimens, having a Type III cadmium plating shall be coated with 0.7 to 1.2 mils (dryfilm thickness) of the primer or enamel intended for use on the plated articles. The coated specimens shall be dried as required for the coating material used plus an additional 24 hours of air drying at room temperature. Dried specimens shall then be immersed in distilled water having a pH of 5.0 to 7.0 at a temperature of 73 °F ($\pm 2^\circ$) for 24 hours. Unsatisfactory water resistance shall be evidenced if any of the following occur for each 48 square inches of surface area tested.

- (a) The appearance of a single blister greater than 3/16 inch long or more than one blister greater than 1/8 inch long.
- (b) The appearance of more than two lines or ringlets of blisters.
- (c) The appearance of 10 or more blisters 1/8 inch or less in length other than the lines or ringlets of (b).

On articles or test specimens having an area less than 48 square inches, a proportionately smaller number of failed areas will be permitted.

4.3.5 Hydrogen embrittlement relief

Hydrogen embrittlement relief tests shall be made by subjecting each of the plated articles in the lot sample to the maximum stress to which it is intended that the articles be exposed in actual service. This stress shall be specified by the MSFC procuring activity and shall be constantly maintained for a minimum period of 240 hours. The failure of any test article in less than 240 hours shall be interpreted as unsatisfactory hydrogen embrittlement relief of the lot represented by that article.

4.4 Retests

Plated articles which have been subjected to a chemical method of plating thickness determination and articles rejected or withdrawn due to defective or deficient plating may be resubmitted after stripping and replating of the individual articles in the first case and of the entire lot in the second case.

5. PREPARATION FOR DELIVERY

5.1 Packaging

Unprotected cadmium-plated articles shall not be packaged in unventilated containers, either together or in contact with electrical equipment, due to the danger of deleterious effects on the cadmium plating from unstable organic electrical insulation. In addition to organic electrical insulation, phenolic resinous substances and others containing unsaturated carbon to carbon linkages, such as oil paints and impregnated paper, etc., cause an abnormal attack on cadmium by setting free formic acid, butyric acid, etc., in the presence of moisture. Corrosion of cadmium plating and steel basis metal has been noted when cadmium plated articles have been packaged in direct contact with container materials such as wood or cardboard. Corrosion has been especially severe when the container materials have become wet or have been stored under conditions of high humidity.

6. NOTES

6.1 Intended Use

This manufacturing specification, developed by the PE Laboratory of

the George C. Marshall Space Flight Center, is intended for use in the cadmium plating of aluminum, copper, and steel alloys for use in space launch vehicle applications.

6.2 Supplementary Treatments

6.2.1 Type II

The primary purpose of chromate (Type II) supplementary treatment is to retard or prevent the formation of white corrosion products on cadmium plating as a result of exposure to stagnant water, high humidity, salt water, marine atmospheres, or to cyclic condensation and drying.

6.2.1.1 Service temperature

Chromate (Type II) finishes are damaged if used on parts continuously exposed to temperatures above 150° F or intermittently exposed for short periods to temperatures of approximately 300° F.

6.2.1.2 Handling

To prevent damage to the soft, gelatinous chromate coating, parts given the chromate (Type II) supplemental treatment by a dipping process must be allowed to air dry for a minimum of 24 hours prior to handling. When an electrochemical process is used, the coating formed is hard and the parts may be handled immediately.

6.2.1.3 Activation

Cadmium-plated surfaces that have become passivated due to exposure to elevated temperatures during hydrogen embrittlement relief treatments may be reactivated prior to chromate (Type II) supplemental treatment. This should be accomplished as soon as possible after embrittlement relief and may be done by immersing the parts for 5 to 15 seconds in an aqueous solution containing 1 percent by volume of the same acid used in the chromating solution into which the part is going to be placed.

6.2.2 Type III

The primary purpose of phosphate (Type III) supplementary treatment is to form a paint base on cadmium plating.

6.3 Safety Precautions

6.3.1 Personnel safety

All necessary safety precautions regarding toxicity and industrial health hazards shall be taken by plating personnel under supervision of the Plating Foreman and Safety Engineer in accordance with MSFC instructions and regulations. Adequate ventilation shall be maintained over the plating tanks and proper precautions taken to assure safe conditions in case of spillage. Suitable gloves, protective clothes, and devices shall be available.

6.3.2 Chemical storage

Plating chemicals shall be stored in secure cabinets or chemical storage rooms which can be maintained in a clean and orderly condition, and which can be easily flushed with water to wash away or neutralize spillage.

6.3.3 Handling containers

All containers employed in the transfer of chemicals shall be washed and cleaned thoroughly immediately after being used.

NOTICE - When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING SPECIFICATION FOR
NICKEL PLATING (ELECTRODEPOSITED)

1. SCOPE

1.1 Scope - This manufacturing specification covers the approved PE Laboratory requirements for nickel plating of components for use in space launch vehicle applications.

1.2 Applicability - The methods and requirements set forth in this specification are applicable to nickel plating of copper and copper alloys, plain carbon steels, low-alloy steels, corrosion or heat resisting steels, nickel alloys, and zinc base alloys.

1.3 Classification - Nickel plating shall be of the following classes and types:

Class 1 Engineering plating plated to specified dimensions or processed to specified dimensions after plating.

Class 2 Decorative plating.

Type I (DS)	- on steel basis metal - 0.002 inch copper plus nickel.
Type II (FS)	- on steel basis metal - 0.00125 inch copper plus nickel.
Type III (KS)	- on steel metal - 0.00075 inch copper plus nickel.
Type IV (QS)	- on steel basis metal - 0.0004 inch copper plus nickel.
Type V (FC)	- on copper-alloy basis metal - 0.0005 inch nickel.
Type VI (KC)	- on copper-alloy basis metal - 0.0003 inch nickel.

Type VII (QC)	- on copper-alloy basis metal - 0.0001 inch nickel.
Type VIII (FZ)	- on zinc alloy basis metal - 0.00125 inch copper plus nickel.
Type IX (KZ)	- on zinc-alloy basis metal - 0.00075 inch copper plus nickel.
Type X (QZ)	- on zinc-alloy basis metal - 0.0005 inch copper plus nickel.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this specification, form a part of this specification.

SPECIFICATIONS

Federal

O-A-76C	Acetic Acid, Glacial, Technical
O-C-275	Chemicals, Photographic "Single Substance", General Specification for
O-O-670	Orthophosphoric Phosphoric Acid, Technical
O-S-61	Salts, Nickel (For) Electroplating and Electrotyping
O-S-809A	Sulphuric Acid, Technical
QQ-A-677	Anode, Nickel
QQ-N-290 (1)	Nickel Plating (Electrodeposited)

Military

JAN-A-183 (2)	Nitric Acid, 42° Baume'
MIL-C-506A (1)	Carbon, Activated, Powdered

MIL-H-11357	Hydrogen Peroxide, 30-Percent Solution
MIL-C-20613	Carbon, Activated, Granular

George C. Marshall Space Flight Center

MS 150.0	Manufacturing Specification for Vapor Degreasing Metallic Surfaces.
MS 150.4	Manufacturing Specification for Electro-polishing of Corrosion Resistant Steel.
10419960	Plating, Cadmium (Electrodeposited), Specification for
MS 101.3	Manufacturing Specification for the Cleaning of Carbon Steel Alloys.

STANDARDS

Military

MS 36043	Hydrochloric Acid
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2.2 Other Publications - The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

American Society for Testing and Materials

A 219-58	Tests for Local Thickness of Electro-deposited Coatings
B 287-61	Standard Method of Acetic Acid - Salt Spray (Fog) Testing

(Copies of these publications may be obtained from the American Society for Testing and Materials, 1916 Race Street, Philadelphia 3, Pa.)

National Bureau of Standards

Journal of Research,
February, 1949

Brenner-Senderoff
Contractometer

(Copies of this publication can be obtained from the Supt. of Documents, Washington, D. C.).

3. REQUIREMENTS

3.1 General - No deviation from the requirements of this manufacturing specification shall be permitted without prior written approval of the MSFC initiating activity.

3.1.1 Base metal forming operations - Unless otherwise specified, plating shall be done after all mechanical operations, brazing or welding, and heat treatments have been performed.

3.1.2 Base metal surface - The surface to be plated shall be free of visible defects that would be detrimental to the appearance or performance of the part. It shall be cleaned, etched, and plated as required to produce the required plating quality.

3.1.3 Preplating stress relief - All uncarburized steel parts having hardnesses greater than Rockwell C-35 shall be stress-relieved prior to cleaning or plating by heating at 375° F ($\pm 10^\circ$) for a minimum of 3 hours.

3.1.3.1 Hardness limitation - No part having a hardness greater than Rockwell C-40 shall be plated without written approval by the initiating activity.

3.1.3.2 Post-plating hydrogen embrittlement relief - Parts, having a hardness greater than Rockwell C-40, shall be heated to 375° F ($\pm 10^\circ$) for 24 hours beginning with 1 hour after the parts have been plated. No plated part of this hardness shall be flexed or stressed prior to baking and pursuant to baking it shall be tested per paragraph 4.3.4.

3.2 Appearance - The nickel deposit shall be smooth, fine grained, adherent, free from blisters, pits, porosity, indications of burning, excessive edge buildup, and other defects. The slight discoloration resulting from baking to relieve hydrogen embrittlement shall not be cause for rejection.

3.3 Thickness

3.3.1 Class 1 plating - Unless otherwise specified, the minimum thickness of Class 1 plating shall be 0.003 inch after all mechanical or other finishing operations have been completed.

3.3.2 Class 2 plating - Unless otherwise specified, minimum plating thickness shall be as specified in Table 4-9.

3.3.3 Plating thickness measurement locations - Plating thicknesses shall be as specified when measured on any visible surface which can be touched by a ball 0.75 inch in diameter.

3.3.4 Plating thickness measurement - The thickness of electrodeposited metal shall be measured microscopically as outlined in A.S.T.M. Standard A219-58.

3.3.4.1 Alternate measurement methods - Suitable magnetic or eddy current measuring instruments (Magne-Gage produced by American Instrument Co., Inc., 8030 Georgia Ave., Silver Spring, Md., or equal) may be used for measuring the plating thickness on steels in lieu of the microscopic method.

3.4 Plating Corrosion Resistance - Nickel-plated shall withstand the acetic acid-salt spray test given in A.S.T.M. Standard B 287-61 for 96 hours without showing signs of corrosion. Plated surfaces, prior to the test, shall be wiped with cotton which has been dipped in a slurry of magnesium oxide. They shall then be rinsed with water and exhibit no water-break. Satisfactory performance shall consist of no more than six corrosion spots per square foot visible to the naked eye or no corrosion spot greater than 1/16 inch in diameter after exposure.

3.5 Plating, Internal Stress - Plating shall be performed under operating conditions which also yield a control of the stress in the electrodeposited metal as measured by the Brenner-Senderoff contractometer or by Erichsen-Cup tests. The stress level shall not exceed that which will yield a satisfactory test result when samples or parts are tested per paragraph 4.3.4.

3.6 Plating, Adhesion - Satisfactory adhesion shall exist if a plated specimen or part, when tested to failure by bending, twisting, or direct stress, exhibits no flaking of the plating when the fracture is viewed under a 4X power magnifier.

TABLE 4-9. MINIMUM PLATING THICKNESSES

Class of Plating	Plated Metal or Metals	Steel Basis Metal				Copper and Copper-Alloy Basis Metal				Zinc Alloy Basis Metal			
		Type				Type				Type			
		DS	FS	KS	QS	FC	KC	QC	FZ(3)	KZ(3)	OZ(4)		
Class 1	Cu Undercoat, in. Nickel, in. Ni + Cu, in. (1)	Optional	----	----	----	2	----	----	0.0004	0.0003	0.0002		
		0.001	0.0006	0.0004	0.0002	0.0005	0.0003	0.0001	0.0005	0.0003	0.0003		
Class 2	Cu Undercoat, in. Ni, in. Ni + Cu, in. (1)	0.002	0.00125	0.00075	0.0004	----	----	----	0.00125	0.00075	0.0005		

- (1) Class 2 platings, including copper undercoat are ordinarily used as undercoat systems for 0.0001-inch electrodeposited chromium.
- (2) Copper alloys containing zinc equal to or greater than 40 percent shall have a copper undercoat of 0.0004-inch minimum thickness for Class I plating and 0.0003-inch for Class 2 plating.
- (3) Copper undercoat may be omitted from Type FZ and Type KZ; the minimum nickel thicknesses shall be as specified for Ni + Cu for these types.
- (4) Copper undercoat may be omitted.

3.7 Materials

- 3.7.1 Anodes - Nickel anodes, cast or rolled, depolarized or carbon type, shall meet the requirements of Specification QQ-A-677.
- 3.7.2 Nickel chloride or nickel sulphate - These salts used in a number of nickel plating baths shall meet the requirements of Specification O-S-61.
- 3.7.3 Boric acid - The boric acid used for bath makeup or additions shall meet the requirements of Specification O-C-275.
- 3.7.4 Sulfamic acid - The acid used for additions shall be equal to DuPont crystalline or granular sulfamic acid possessing a purity of 99 percent acid.
- 3.7.5 Nickel sulfamate - This liquid solution, of 48 degrees Baume' and possessing a concentration of 98 to 99 percent, shall be equal to that supplied by Barrett Chemical Products Company of Shelton, Connecticut.
- 3.7.6 Hydrochloric acid - The acid used in the nickel strike bath shall meet the requirements of Standard MS 36043.
- 3.7.7 Nickel carbonate - Nickel carbonate used for chemical reaction with sulfamic acid to produce nickel sulfamate shall be of a technical grade. The nickel carbonate added for pH correction shall also be of a technical grade.
- 3.7.8 Nickel ammonium sulphate - This double salt of nickel shall meet the requirements of Specification O-S-61.
- 3.7.9 Zinc sulphate - The zinc sulphate used shall be either a purified or reagent quality.
- 3.7.10 Sodium thiocyanate - The sodium thiocyanate shall meet the requirements of Specification O-C-275.
- 3.7.11 Demineralized water - Filtered water, possessing a minimum resistivity of 50 000 ohms, shall be used where required.
- 3.7.12 Addition agents - Additives to nickel plating baths for brightening, leveling of the deposit, stress reduction and nonpitting shall be judged for acceptability by the results of Hull Cell tests and by their effect on internal stress covered in paragraph 3.5 of this specification.

3.7.13 Sulphuric acid - Sulphuric acid used to adjust the pH value of the bath and for pickling shall meet the requirements of Specification O-S-809A.

3.7.14 Activated carbon - The activated carbon used in mechanical filters or as a bath additive shall meet the requirements of Specification MIL-C-20613 or MIL-C-506A (1).

3.7.15 Hydrogen peroxide - The hydrogen peroxide used as a "non-pitter" addition shall meet the requirements of Specification MIL-H-11357.

3.7.16 Wetting agents - These additions for relief of pitting shall be judged for merit by platinum tensiometer results and their ability to eliminate pitting.

3.8 Equipment

3.8.1 Anode bags - Dynel woven in a twill weave, having a thread count of 72×48 and a weight of 6.76 ounces per square yard, shall be used for manufacturing anode bags. The bags shall be desized before use and have a length about 5 inches longer than the anode. The bags may be further strengthened by dipping the bottom ends in a synthetic resin. These loose-fitting bags shall be tied above the solution and around the hook above the anode.

3.8.2 Filter - Any filter that will turn over the solution once an hour; that is lined with lead, rubber, or suitable plastic; and that utilizes activated carbon, is acceptable. A lead lining shall not be used when the chloride content of the bath exceeds 10 ounces per gallon.

3.8.3 Tanks

3.8.3.1 Nickel-plating tank - A steel tank of welded construction, lined with rubber, synthetic plastic, or acid resistant brick may be used.

3.8.3.2 Storage tank - An auxiliary tank of a construction similar to the nickel-plating tank may be provided for efficient solution make up or occasional bath purification.

3.8.4 Temperature control - Steam coils made of tantalum or heat exchangers made of Duriron may be used for heating the bath. Lead must not be used in sulfamate baths or in high chloride Watts bath.

3.8.5 Agitation - Agitation may be by air or by mechanical means. Air should not be supplied from high pressure plant systems. Danger of oil contamination from these systems is great. For air agitation, the tank shall be

equipped with a spider made of plastic pipe so positioned and drilled that the agitation will be vigorous and uniform throughout the tank. Air should be supplied by a low pressure blower that has a filter installed on the blower's inlet. The blower specification should be as follows for 1/36 inch deep bath; outlet pressure 3 psi and a minimum discharge volume of 1.5 C.F.M. for each square foot of tank surface.

NOTE: Air agitation should not be used in nickel baths to which organic brighteners have been added as it will cause excessive foaming, decomposition of the brightener, and pitting of the plating deposit.

3.8.6 Racks - Racks must hold work pieces firmly to preserve electrical contact because work pieces are exposed to the air agitation in the bath. The weight of racks plus work pieces must be ample to resist swinging in the agitated solution. Racks should also be coated with plastisol.

3.8.7 Power supply - A selenium rectifier or motor generator set capable of generating a direct current of sufficient magnitude for the type of bath and the area of parts being plated at a maximum voltage of 20 shall be equally acceptable.

3.9 Procedures

3.9.1 Pretreatments (prior to plating)

3.9.1.1 Steels of less than 7 percent alloying elements - Components made of these steels shall be cleaned for nickel plating per Specification MS 101.3.

3.9.1.2 Corrosion or heat resistant steels - Components made of these steels shall be cleaned for nickel plating per Specification MS 150.4. The electropolishing operation shall be optional.

3.9.1.3 Copper and copper alloys - Components made of these materials shall be cleaned as follows:

A. Scale removal - When heavy scale such as results from heat treatment must be removed, parts shall be pickled in an aqueous solution of 5 to 10 percent by volume of sulfuric acid at room temperature for 1 to 15 minutes.

B. Degreasing - Parts shall be degreased per Specification MS 150.0.

C. Alkaline degrease - Parts shall be immersed in an aqueous solution of Wyandotte B.N., or equal, at a concentration of 4 ounces per gallon, at a temperature of 190 to 210° F for 2 to 20 minutes depending on the degree of soil.

D. Rinse - Parts shall be rinsed by dipping in cold water.

E. Electroclean - Parts shall be electrocleaned in an aqueous solution of Wyandotte B.N. at a concentration of 4 to 6 ounces per gallon, at a temperature of 150 to 180° F, at a current density of 20 to 50 amperes per square foot for 1 to 3 minutes cathodically, and for 5 to 10 seconds anodically.

F. Bright dip - To remove tarnish and stain as well as to smoothen the surface, parts shall be immersed in a bath composed of 20 percent by weight nitric acid, 25 percent by weight glacial acetic acid, 54.5 percent by weight phosphoric acid, and 0.5 percent hydrochloric acid. The bath shall be operated at 190° F ($\pm 5^\circ$) and the time of immersion may be up to 5 minutes.

G. First rinse - Parts shall be rinsed by immersion in water at 150° F ($\pm 10^\circ$).

H. Second rinse - Parts shall be immersed in a cold flowing rinse and transferred quickly to the plating bath.

3.9.2 Plating bath compositions and conditions - Plating bath compositions and operating conditions shall be as specified in Table 4-10.

3.9.3 Plating solution makeup

3.9.3.1 Watts type bath

- In a clean, storage tank dissolve in water at 130 to 140° F the calculated nickel sulfate and nickel chloride. Add one pint of 30 percent hydrogen peroxide and digest for 1 hour while stirring.
- Add a slurry of nickel carbonate in water until the pH reaches 4.8 to 5.0.
- Add 1/2 to 3 pounds of activated carbon as a slurry in water to each 100 gallons of plating solution and agitate the solution for several hours at 130 to 140° F.

TABLE 4-10. PLATING BATH COMPOSITIONS AND CONDITIONS

Bath Type & Composition	1. Watts Type (oz/gal)	2. Watts Type High Cl (oz/gal)	3. Sulfam- ate (oz/gal)	4. Black Nickel Oxide (oz/gal)	Nickel Strike (oz/gal)
NiSO ₄ ·6H ₂ O	40.	32.			
NiCl ₂ ·6H ₂ O	6.-8.	12.	0.44		32.0
Ni(SO ₃ NH ₂) ₂			60		
NiSO ₄ (NH ₄) ₂ SO ₄ ·6H ₂ O				8	
ZnSO ₄ ·7H ₂ O				1	
NaCNS				2	
HCL					10
H ₃ BO ₃	4-5.5	4-5	2-5		
H ₂ O ₂	5-10	5-10			
pH	3.5/4.8	2.0/2.5	3.0/5.0	5.5/6.0	
° F	115-140	130-140	100°/140°	75°-120°	75°-85°
A/Ft ²	10-30	10-60	50-300	2-20	50-150
Volts	6-12	6-12	6-9	1-2	6.
Anode to Cathode Area	1:1	1:1	1:1	1:1	

NOTE 1 Baths 1 and 2 used for dull nickel (0.1-2.0 Mils+). For semi or full bright 0.1-2.0 mils use addition agents. For decorative or protective, copper and/or nickel-chromium on most metals use No. 1 or 2. For corrosion protection use No. 1 or 2. For building up worn parts use No. 1 or 2. The nickel strike is used prior to precious metal plating or any metal plating.

NOTE 2 The addition of saccharin to bright Watts bath reduces to a low value the internal stress in the coating. Bright stressed deposits corrode easier and when scratched fail by stress-corrosion cracking.

NOTE 3 Bath No. 3 yields hard, low stress deposits.

- Allow to settle and filter supernatant liquid into the plating tank.
- Dissolve the required amount of boric acid in warm water and add to the bath.
- Insert nickel anodes and dilute to plating volume.
- Adjust the pH to 3.0 electrometrically using sulphuric acid.
- Electrolyze the bath for several hours at a low current density (2-5 amperes per square foot), at a temperature of 130° F, and with as large a cathode area as possible.
- Adjust pH to 4 electrometrically. Use nickel carbonate to raise it and sulphuric acid to lower it.
- Add the necessary addition agents for non-pitting, brightening and stress reduction. Brightener No. 2-RL, Non-Pitter No. 22, and Brightener No. 7 manufactured by Udyllite Corporation of Detroit, Michigan may yield acceptable results per paragraph 3.5.

3.9.3.2 Sulfamate bath - The solution given in Table 4-10, bath No. 3, can be prepared from available concentrates of nickel sulfamate diluted to the proper concentration, and subsequent purification by the usual high pH and peroxide treatment to remove iron. This is followed by carbon filtration and low current density electrolytic "dummying" to remove copper, zinc, and other metallics. The wetting agent and boric acid are added and the pH is adjusted to the correct range with sulfamic acid. Continuous filtration with activated carbon is not recommended as it will remove the stress reducer (SNSR, obtained from Barrett Chemical Products Company, Bridge Street, Shelton, Connecticut) and the wetting agent that may be added.

3.9.3.3 Black nickel bath - This bath is prepared from the constituents in Table 4-10 by dissolving the ingredients in water at 120° F ($\pm 10^\circ$). It does not need filtering or a low-current density electrolysis for purification.

3.9.3.4 Nickel strike bath - This bath is prepared by first dissolving the nickel chloride in 140° F ($\pm 10^\circ$) water and subsequently adding the required hydrochloric acid.

3.9.4 Plating procedure

3.9.4.1 General - Consideration shall be given to deposit specification, shape of part, type and method of stop-off, method of holding work, size of the rack or holder, total current required, and compatibility with other parts to be plated.

3.9.4.2 Immersion - Properly racked parts shall be immersed before applying the voltage. Parts shall remain in the bath for the required time before power is interrupted. If the coating thickness is inadequate, it shall be returned immediately to the bath to prevent layering of the second nickel deposit. Tarnished parts shall be cathodically activated in a 10-percent by weight hydrochloric acid solution or by swirling in a nickel strike solution for less than 1 minute. Parts shall not be immersed in the plating bath with live contacts to prevent uneven current distribution and possible burning.

3.9.5 Rinse - Parts shall be rinsed by immersion in hot water at 150° F ($\pm 10^\circ$) followed by a cold running rinse.

3.9.6 Post-plating bake - Parts shall be processed per paragraph 3.1.3.2 of this specification.

3.9.7 Electropolishing - When nickel plating is to be electropolished prior to chromium plating, the finer grained deposits of the semi-bright Watts bath or of the sulfamate bath shall be used to avoid a smoky appearance of the chromium plating in bright sunlight.

3.9.8 Plating of corrosion resistant steel and nickel alloys - Because these materials tend to self-passivation, handling between activating and plating must be rapid. These materials may require a two-tank treatment as an alternate to that indicated in paragraph 3.9.1.2.

3.9.8.1 Acid etch - A bath composed of 50 percent by volume hydrochloric acid at room temperature, an immersion time up to 1.5 minutes, the work made anodic, and a current density of 20 amperes per square foot shall be used.

3.9.8.2 Nickel-strike - In the second tank the work is again made anodic for 15 seconds before the cathodic phase.

3.9.8.3 Rinse - Rinsing in cold water should be thorough, followed by rapid transfer to the first coat plating tank.

3.9.9 Purification of plating solutions - Because nickel baths are usually prepared from technical grade salts, impurities must be eliminated. The methods used shall be:

3.9.9.1 Electrolytic - This method was indicated in paragraph 3.9.3.1.

3.9.9.2 High pH - For the removal of iron or trivalent chromium, heat the bath to 150° F. Raise the pH to 5 minimum with a slurry of nickel carbonate. Add ferrous sulphate (10 ounces per 100 gallons) and oxidize the iron to the ferric state with an addition of hydrogen peroxide (10 fluid ounces per 100 gallons). Add ferrous chloride to the nickel strike bath. Agitate the bath and allow to settle overnight. Pump off the liquid through a filter charged with filter aid, do not disturb the precipitate in the tank.

3.9.9.3 Activated carbon - This treatment removes organic impurities. It should not be done in the plating tank as carbon will adhere to the walls and later cause roughness in the deposit.

3.9.10 Stripping of nickel plate

3.9.10.1 From steels - Immersion at room temperature in a concentrated nitric acid solution containing 0.1 fluid ounce of hydrochloric acid per gallon of nitric acid used, or anodic pickling in a 60 percent by volume sulphuric acid solution using lead cathodes is effective. A 2-volt battery is adequate for the necessary current in the anodic method.

3.9.10.2 From copper alloys - Anodic, room temperature pickling, at 6 to 12 volts, in a solution of 2 ounces of hydrochloric acid per gallon of water with carbon cathodes is effective.

4. QUALITY ASSURANCE PROVISIONS

4.1 General - To effectuate adequate control over the quality of plating, routine analyses shall be made of the composition of the plating bath and other processing baths. Verification of analyses and tests, in the form of recorded data, shall be maintained. The results of all routine tests for stress, adhesion, thickness, and corrosion shall also be recorded.

4.2 Sampling

4.2.1 Lot - For the purposes of this specification, a lot is defined as a group of plated articles of the same size, the same shape, having the same type and class of nickel plating, and submitted for inspection at the same time.

4.2.2 Sampling for visual inspection and for non-destructive tests of plating thickness - A sample shall be drawn from each inspection lot by taking at random the percentage of articles specified in Table 4-11. These articles shall then be subjected to both the visual inspection and the plating thickness determination outlined in paragraphs 4.2.2.1 and 4.2.2.2, respectively.

4.2.2.1 Visual examination - All sample articles selected in paragraph 4.2.2 shall be visually examined to assure conformance to the requirements of paragraph 3.2. If the number of non-conforming articles exceeds the acceptance number for that size sample (see Table 4-11), the lot represented by that sample shall be rejected.

4.2.2.2 Thickness determination - All sample articles selected in paragraph 4.2.2 shall have their plating thickness measured by a non-destructive method at several locations on each article where the plating is expected to be of minimum thickness. An article shall be considered to be nonconforming if any measurement fails to meet the specified minimum thickness. If the number of nonconforming articles in any sample exceeds the acceptance number for that size sample (see Table 4-11), the lot represented by that sample shall be rejected.

4.2.3 Sampling for destructive tests of plating thickness, adhesion, and salt spray resistance - A random sample of the size specified in Table IV, but composed of different articles than those selected for non-destructive tests in paragraph 4.2.2, shall be selected from each inspection lot and eight separately plated test specimens shall be prepared to represent each inspection lot.

4.2.3.1 Thickness tests - Each article of the sample selected in 4.2.3 shall be tested in several locations at which the plating would be expected to be a minimum. If the plating thickness at any location on any article is less than the specified thickness, the lot represented by that article shall be rejected.

4.2.3.2 Salt spray resistance - For Class I nickel plating, four of the separately plated test specimens prepared in paragraph 4.2.3 shall be tested in lieu of plated articles which comprise the inspection lot. The failure of any test specimen in less than the time specified in paragraph 3.4 shall result in the rejection of the lot represented by that test specimen.

4.2.3.3 Adhesion - All articles used for plating thickness tests, if of suitable size and form, shall be used as specimens for the adhesion tests. If

TABLE 4-11. SAMPLING FOR NONDESTRUCTIVE TESTS

ITEMS IN INSPECTION LOT (NUMBER)	ITEMS IN SAMPLE (PERCENT OF INSPECTION LOT)		ACCEPTANCE NUMBER (MAXIMUM ALLOWABLE NUMBER OF ITEMS NONCONFORMING)
	Visual, 4.2.2.1	Thickness, 4.2.2.2	
1 to 6	100	100	0
7 to 15	100	100	0
16 to 40	100	100	0
41 to 110	100	60	0
111 to 300	60	40	1
301 to 500	30	20	1
501 and over	20	15	2

the articles used for plating thickness tests are not of suitable size and form, an equal number of additional articles shall be selected or separately plated test specimens used. Failure of any test specimen shall result in the rejection of the lot represented by that test specimen.

4.2.3.4 Hydrogen embrittlement relief - A random sample of the size specified in Table 4-12 shall be selected from each lot of plated articles and tested for hydrogen embrittlement relief in the manner specified in paragraph 4.3.4. Failure of any of the articles tested shall result in the rejection of the lot represented by that article.

4.3 Test Methods

4.3.1 Thickness - Thickness determinations shall be made by the microscopic method, or by an instrument or electrochemical method approved by the MSFC initiating activity. For referee tests, the microscopic method shall be used. Measurement of minimum plating thickness on bolts shall be made on the shank adjacent to the threat, or on the head if the shank is completely threaded.

4.3.1.1 Microscopic test

A. Specimens - Specimens for microscopic measurements shall consist of cross-sections of plated articles, or test specimens, suitably mounted, ground, polished, and etched to differentiate the nickel plating from the basis metal.

B. Metallurgical microscope - The plating thickness of prepared specimens may be measured by means of a metallurgical microscope equipped with a filar micrometer ocular which has been calibrated against a standard scale to an accuracy of ± 2 percent.

C. Metallographic microscope - The plating thickness of prepared specimens may be measured by means of a metallographic microscope by projecting the image of the specimen on the ground-glass focusing plane of the camera at a known magnification. The actual plating thickness can then be easily determined by measuring the plating thickness of the projected image in inches, and dividing this measured value by the known image magnification.

TABLE 4-12. SAMPLING FOR DESTRUCTIVE TESTS

ITEMS IN INSPECTION LOT (NUMBER)	ITEMS IN SAMPLE (NUMBER)	ACCEPTANCE NUMBER (MAXIMUM ALLOWABLE NUMBER OF ITEMS NONCONFORMING)
1	0	0
2 to 5	1	0
6 to 25	3	0
26 to 100	4	0
101 to 300	6	0
301 to 5000	10	0
5001 and over	15	0

4.3.2 Adhesion

4.3.2.1 Scrape method - Adhesion may be determined by scraping the surface of the plated article, or test specimen, to expose the basis metal. The junction of the plating and the exposed basis metal shall then be examined at four diameters magnification. Any evidence of flaking, blistering, or separation between the plating and the basis metal shall be interpreted as unsatisfactory adhesion.

4.3.2.2 Bend method - Adhesion may be determined by clamping the plated article, or test specimen, in a suitable holding device and rapidly bending at room temperature through an angle of 180 degrees, around a diameter equal to the thickness of the specimen, until plating rupture occurs. If the edge of the ruptured plating can be peeled back or if separation between the plating and the basis metal can be seen at the point of rupture when examined at four diameters magnification, adhesion shall be interpreted as unsatisfactory.

4.3.3 Salt spray resistance - Salt-spray resistance tests shall be made in the manner specified in Method 812 of Federal Test Method Standard Number 151a.

4.3.4 Hydrogen embrittlement relief - Hydrogen embrittlement relief tests shall be made by subjecting each of the plated articles in the lot sample to the maximum stress to which it is intended that the articles be exposed in actual service. This stress shall be specified by the MSFC Procuring Activity and shall be constantly maintained for a minimum period of 240 hours. The failure of any test article in less than 240 hours shall be interpreted as unsatisfactory hydrogen embrittlement relief of the lot represented by that article.

4.4 Retests - Plated articles which have been subjected to a chemical method of plating thickness determination and articles rejected or withdrawn due to defective or deficient plating may be resubmitted after stripping and replating of the individual articles in the first case and of the entire lot in the second case.

4.5 Nickel Plating Solution Analyses

4.5.1 Determination of nickel

- o Pipette a 10 cc sample into a 200 cc electrolytic beaker.

- Add concentrated ammonia (NH_4OH , Sp. Gr. 0.897) until the solution turns distinctly blue, and then add an excess of 20 ml.
- Add 5 grams of ammonium sulphate.
- Dilute to about 150 ml.
- Dip the platinum gauze in ethyl alcohol, touch to a blue flame from a Fischer burner, allow to burn off away from the flame, and then hold in the flame until red hot.
- Weigh the cathode as soon as cool.
- Fasten the cathode in the electrolytic apparatus so that about $\frac{1}{2}$ inch of gauze is above the solution level.
- Electrolyze until all the nickel in solution is deposited. Use $\frac{1}{2}$ ampere overnight or 2 amperes with agitation for about 2 hours.
- Remove the cathode, washing all the solution back into the beaker, and save the solution for the boric acid determination.
- Dip the cathode in ethyl alcohol, touch to a blue flame, and allow to burn off while waving gently back and forth.
- Weigh the cathode when cool.
- Calculate as follows - oz/gal. Weight of nickel in grams \times 13.3 = oz/gal. Nickel (Ni)

4.5.2 Determination of boric acid

- Pipette a 1-ml sample into a 250-ml Erlenmeyer flask.
- Add 3 to 5 drops of a bromthymol blue-bromcresol purple indicator made by dissolving 1 gram of bromthymol blue and 5 grams of bromcresol purple in 500 ml of ethanol.
- Add approximately 5 grams ($\frac{1}{6}$ oz) of mannitol and gently shake until a thick paste is formed.

- Fill a 50-ml burette with a standard sodium hydroxide solution and titrate the sample until the color changes from green to blue.
- Read the burette. The ml of sodium hydroxide solution used multiplied by the proper factor for standard sodium hydroxide solution gives the ounces per gallon of boric acid in the plating bath.

Calculate the factor as follows:

$$\frac{\text{*Wt. of KHC}_8\text{H}_4\text{O}_4 \times 0.303 \times 1000}{\text{Ml of Standard NaOH} \times 7.5} = \text{Factor in}$$

ounces per gallon for boric acid (H_3BO_3).

* Wt. in grams of potassium acid phthalate ($\text{KHC}_8\text{H}_4\text{O}_4$) used in preparing standard NaOH solution.

4.5.3 Determination of chloride

- Pipette a 5 cc sample into a 250 cc Erlenmeyer flask.
- Dilute to 75 to 100 cc.
- Add three drops of methyl orange indicator. If the solution is pink rather than yellow, add sodium hydroxide (10 gm/liter), drop by drop, until it just turns yellow. (High results are obtained if the solution is above a pH of 4.)
- Add 1 cc sodium chromate (20 gm/liter).
- Allow standard silver nitrate (0.1N) to run from a burette until the solution just turns a light buff color.
- Calculate -

$$\text{cc } 0.1\text{NAgNO}_3 \times 0.0948 = \text{oz/gal chloride.}$$

4.5.4 Determination of NaCNS

- Pipette 10 cc into 250 cc Erlenmeyer flask.

- Dilute by adding 100 cc distilled water then add concentrated H_2SO_4 until acid by litmus test.
- Add 15 cc ferric alum solution which is prepared by dissolving 10 gm of ferric ammonium sulphate and 0.25 ml of concentrated nitric acid in distilled water. Dilute to make 500 cc.
- Titrate with standard AgNO_3 solution until red color disappears.
- Read burette - Number of cc of standard AgNO_3 used multiplied by the proper factor for the standard AgNO_3 solution equals the ounces of NaCNS per gallon of plating bath.
- Calculation of factor for standard AgNO_3 solution:

$$\frac{\text{Wt of KCl} \times 1.088 \times 100}{\text{Ml of STD. AgNO}_3 \times 7.5} = \text{Factor for use in calculating oz/gal of NaCNS}$$

4.5.5 Determination of zinc

- Pipette a 10 cc sample into a 250 cc beaker.
- Add 20 cc of 3 percent NaCN. Solution should be clear. If not add more until it clears. Heat to 200-210°F.
- While stirring add 25 cc of Na_2S and allow to settle. Na_2S solution is made by dissolving 150 gm of $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ in sufficient water to make a liter.
- Filter while hot and wash twice with water containing a few drops of the sodium sulfide solution.
- Transfer precipitate and filter paper back to beaker in which precipitation was made then add a few crystals of sodium sulfide and 10 cc of concentrated HCl.
- Boil to expel H_2S gas.
- Add 150 cc of distilled water and heat to 200 to 210°F.
- Titrate the above with standard potassium ferrocyanide solution, maintaining the temperature above 170°F, until a drop

of solution from the beaker gives a faint brown coloration when transferred to a drop of uranium acetate on a porcelain spot plate. The uranium acetate solution is made by dissolving 22 g of uranyl acetate plus 10 ml of concentrated acetic acid in 500 ml of distilled water.

- The burette reading multiplied by the factor for the standard potassium ferrocyanide solution gives the ounces of zinc per gallon of the plating bath.
- Calculate factor for standard $K_4Fe(CN)_6$ as follows:

$$\frac{\text{Wt of zinc in 10 cc aliquot} \times 100}{\text{Ml of standard } K_4Fe(CN)_6 \cdot 3H_2O \times 7.5} =$$

Factor in oz/gal for metallic zinc.

4.5.6 Sulfamic acid determination - A rapid gasometric method of determining sulfamic acid by measuring the volume of released nitrogen gas that is generated by the reaction of sodium nitrite with sulfamic acid is given in Analytical Chemistry, Volume 23, 1951, page 1016.

5. PREPARATION FOR DELIVERY

Not applicable to this specification.

6. NOTES

6.1 Intended Use - This manufacturing specification developed by the PE Laboratory of George C. Marshall Space Flight Center is intended for use when nickel plating is performed on space vehicle components.

6.2 Important Considerations

6.2.1 Ammonium salts - Ammonia and ammonium salts must be kept out of nickel baths. They cannot be removed.

6.2.2 Grease, oil and buffing compound - See that such materials are removed in pretreatment processes, either by use of the overflowing tank feature or by removing them from the surface of the bath by blotting with clean newspapers.

6.2.3 Fallen parts - Do not allow parts to accumulate in the bottom of the tank. They will dissolve and contaminate the bath.

6.2.4 Copper flash - Take care that the copper plating solution used prior to nickel plating is kept in good condition. Rough copper deposits mean rough nickel coatings. Also rinse very thoroughly afterwards to avoid copper contamination of the nickel bath.

6.2.5 Filtration - Remember that continuous filtration will remove desirable and undesirable organic materials.

NOTICE - When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING SPECIFICATION FOR
CHROMIUM PLATING (ELECTRODEPOSITED)

1. SCOPE

1.1 Scope - This manufacturing specification covers the approved PE Laboratory requirements for chromium plating of components for use in space launch vehicle applications.

1.2 Applicability - The methods and requirements set forth in this specification are applicable to chromium plating of carbon steels, low-alloy steels, corrosion or heat resisting steels, aluminum and aluminum alloys, and copper and copper alloys.

1.3 Classes and Types - Chromium plating shall be of the following classes and types, as specified:

Class 1 - Decorative plating.

Type I - Bright finish

Type II - Satin finish

Class 2 - Hard (for engineering use).

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this specification, form a part of this specification.

SPECIFICATIONS

Federal

O-A-76c	Acetic Acid, Glacial, Technical
O-H-765a	Acid, Hydrochloric (Muriatic), Technical Grade

O-C-303	Chromium Trioxide, Technical (Chromic Acid)
O-E-760b(2)	Ethyl Alcohol (Ethanol); Denatured Alcohol and Proprietary Solvent
O-N-350	Nitric Acid, Technical
O-O-670	Orthophosphoric (Phosphoric) Acid, Technical
O-S-809a	Sulphuric Acid, Technical
QQ-C-320(1)	Chromium Plating (Electrodeposited)
QQ-N-290(1)	Nickel Plating (Electrodeposited)

Military

MIL-C-506A(1)	Carbon, Activated, Powdered
MIL-S-13165	Shot Peening of Ferrous Metal Parts
MIL-C-14550	Copper Plating (Electrodeposited)
MIL-C-20613	Carbon, Activated, Granular

George C. Marshall Space Flight Center

MS 100.2	Manufacturing Specification for The Deoxidation of Aluminum Alloys
MS 101.3	Manufacturing Specification for The Cleaning of Carbon Steel Alloys
MS 120.1	Manufacturing Specification for Nickel Plating (Electrodeposited)
MS 150.0	Manufacturing Specification for Vapor Degreasing Metallic Surfaces
MS 150.4	Manufacturing Specification for Electro- polishing of Corrosion Resistant Steel

STANDARDS

Federal

151a

Metals; Test Methods

2.2 Other Publications- The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

American Society for Testing and Materials

A219-58	Tests for Local Thickness of Electro-deposited Coatings
B177-55	Chromium Plating on Steel for Engineering Use
B253-53	Preparation of and Electroplating on Aluminum Alloys
B368-61T	Method of Copper-Accelerated Acetic Acid Salt Spray (Fog) Testing (Cass Test) Tentative

(Copies of the publications listed above may be obtained from the American Society for Testing and Materials, 1916 Race Street, Philadelphia 3, Pa.).

National Bureau of Standards

Journal of Research February, 1949	Brenner - Senderoff Contractometer
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(Copies of the publication listed above may be obtained from the Supt. of Documents, Washington 25, D.C.).

3. REQUIREMENTS

3.1 General - No deviation from the requirements of this manufacturing specification shall be permitted without prior written approval of the MSFC initiating activity.

3.2 Equipment

3.2.1 Tanks - A steel tank of welded construction, lined with vinyl, lead, or acid resistant brick may be used at usual temperatures. Other organic coatings fail. Special high concentrations and temperatures require acid brick set over the lining in a silicate cement.

3.2.1.1 Storage tank - An auxiliary tank of a construction similar to the plating tank may be provided for solution make-up or occasional bath purification.

3.2.2 Temperature control - Steam coils or heat exchangers made from lead or Duriron may be used for heating the bath.

3.2.3 Agitation - Agitation may be by air or by mechanical means. Air should not be supplied from high pressure systems as danger of oil contamination from these systems is great. For air agitation, the tank shall be equipped with a spider made of plastic pipe so positioned and drilled that the agitation will be vigorous and uniform throughout the tank. Air should be supplied by a low pressure blower that has a filter installed on the blower inlet. The blower specification should be as follows for a 36-inch deep bath; outlet pressure 3 psi and a minimum discharge volume of 1.5 cubic feet per minute for each square foot of tank surface.

3.2.4 Racks

3.2.4.1. The parts to be plated may be assembled with the rack (or fixture) at any convenient stage in the preparatory process.

3.2.4.2 While the general principles of good racking as used in other plating processes apply, the application of the higher current densities necessary for chromium plating and the desirability of securing coatings of uniform thickness and quality on desired areas require rack construction methods that are much more precise. The design of racks for chromium plating of steel for engineering use shall provide for the following:

- Sufficient current carrying capacity of both cathode and anode circuits to all parts on the rack.
- Positive contacts to the parts to be plated and to the tank rods.
- Uniformity of current distribution on the parts to be plated. This often requires anodes of special shapes conforming to the shape of the part or area being plated.

- The use of "thieves", "robbers", or "guards", which are auxiliary metallic conductors placed near points of abnormally high current density to attract the current away from such points; and "shields" which are parts made of nonconductive materials placed in the path of the current where it tends to concentrate unduly.
- The use of "masks" made of rigid materials placed against a surface to prevent local plating, and "stopoffs" which are especially compounded lacquers or waxes for the protection of such surfaces.
- The use of nonconducting plugs in holes not requiring plating to produce a sharp edge without grooves around the periphery of the holes.
- The use of materials sufficiently insoluble and noncontaminating in the solutions to have the desired life.
- Placement of the parts in such positions that gas from the parts, "thieves", and anodes escapes freely and does not become entrapped so as to prevent plating on areas which should be plated.

3.2.5 Anodes—A lead alloy containing 6 percent antimony is most commonly used in the construction of anodes. Lead alloys containing 7 percent tin or 1 percent silver may be used alternatively. Chemical grade lead is satisfactory where hardness and rigidity are no object. Anodes of composite structure, comprising lead-sheathed steel or copper, may be used when indicated by the requirement for strength or conductivity. Where platinum is not available for internal plating of small holes, iron or steel rods may be used at the expense of contamination of the solution with iron.

3.2.6 Power supply - A selenium rectifier or motor generator set capable of generating a direct current of sufficient magnitude for the type of bath and the area of parts being plated, at a maximum voltage of 20, shall be provided.

3.3 Materials - Materials such as basis metals, chemicals, addition agents, wetting agents, water, and cleaning agents shall produce coatings which meet the requirements of this specification and which will have no deleterious effect on the corrosion resistance of the basis metal.

3.3.1 Basis metal - The basis metal shall be free from visible defects that will be detrimental to the appearance or the performance of the plating.

3.3.2 Chemicals - The basic chemicals used in the chromium plating solution, the metallographic etching agent, the addition agents, the wetting agents, and the cleaning agents shall meet the requirements of the applicable specification referenced in paragraph 2.1.

3.3.3 Addition agents - Additives to chromium plating baths for leveling of the deposit, stress reduction, and non-pitting shall be judged for acceptability by the results of Hull Cell tests and by their effect on internal stress covered in paragraph 3.7.

3.3.4 Wetting agents - Additions for relief of pitting shall be judged for merit by platinum tensiometer results and their ability to eliminate pitting.

3.3.5 Demineralized water - Filtered water, possessing a minimum resistivity of 50 000 ohms, shall be used in the chromic acid solution bath and in rinse tanks.

3.4 General Procedure

3.4.1 Plating solution makeup

3.4.1.1 In a clean storage tank, dissolve in water at 130 to 140°F the calculated chromic acid (33.0 oz per gallon, formula No. 1, Table 4-13). Digest solution for 1 hour while stirring.

3.4.1.2 Add the sulphuric acid (0.33 oz per gallon, formula No. 1, Table 4-13). Maintain the bath ratio, $\text{CrO}_3/\text{H}_2\text{SO}_4$ at 100:1.

3.4.1.3 Allow to settle and then filter solution into plating tank.

3.4.1.4 Insert lead alloy anodes, immerse to plating depth.

3.4.1.5 Electrolyze the bath for several hours at a low current density, 0.7 to 0.8 [amperes per square foot (asf)], at a temperature of 130 to 140°F, and with as large a cathode as possible.

3.4.1.6 Using a hydrometer, check the specific gravity or Baume of the bath at 60°F ($\pm 2^\circ$). The ounces of chromic acid per gallon of solution, corresponding to the specific gravity or Baume determined, will be as shown in Table 4-14.

TABLE 4-13. CHROMIUM PLATING BATH
COMPOSITIONS AND OPERATING CONDITIONS

Composition	Bath 1	Bath 2
Chromic acid, CrO_3 , oz./gal.	33.0	53.0
Sulfuric acid, H_2SO_4 , oz./gal.	0.33	0.53
Bath ratio, $\text{CrO}_3/\text{H}_2\text{SO}_4$	100:1	101:1
Conditions: (a)		
Temperature, °F	110-120	110-120
Current density, asi	0.7-1.5	0.7-1.5
Agitation, cathode	Optional	Optional
Cathode efficiency, % (approximate)	13	13
Ratio anode-to-cathode area	1:1-2:1	1:1-2:1
Anodes, lead, usually an alloy of 6% antimony, tin or tellurium		
Filtration: Not usual		
Volts:	6-12	6-12

(a) Temperatures from 70 to over 150 F with variations in cathode current densities from 1.5-8.0 asi and cathode efficiencies between 10 and 20% are possible. For bright deposits the exact values must be carefully controlled and related.

TABLE 4-14. SPECIFIC GRAVITY AND BAUME OF CrO_3 SOLUTIONS

Sp. Gr. at 59°F 39.2°F	°Be at 60°F	Chromic Acid. oz/gal	Sp Gr. at 59°F 39.2°F	°Be at 60°F	Chromic Acid oz/gal
1.01	1.44	2.0	1.18	22.10	34.4
1.02	2.94	3.9	1.19	23.20	36.4
1.03	4.22	5.8	1.20	24.20	38.4
1.04	5.58	7.6	1.21	25.20	40.3
1.05	6.90	9.5	1.22	26.20	42.3
1.06	8.20	11.4	1.23	27.10	44.2
1.07	9.50	13.4	1.24	28.10	46.2
1.08	10.07	15.3	1.25	29.00	48.2
1.09	12.00	17.3	1.26	29.99	50.2
1.10	13.00	19.1	1.27	30.80	52.2
1.11	14.40	21.0	1.28	31.70	54.5
1.12	15.50	22.9	1.29	32.60	56.5
1.13	16.70	24.8	1.30	33.50	58.7
1.14	17.80	26.8	1.31	34.30	60.7
1.15	18.90	28.8	1.32	35.20	62.7
1.16	20.00	30.6	1.33	36.00	64.8
1.17	21.10	32.6	1.34	36.80	67.0

3.4.1.7 Sulphate content of the new bath may be checked in the manner outlined in paragraph 4.5.2.

3.4.2 Typical chromium plating bath analysis - Plating bath compositions and operating conditions shall be as specified in Table 4-13.

3.4.3 Fabrication of components - Unless otherwise specified, chromium plating shall be applied after all basis metal heat treatments and mechanical operations such as machining, brazing, welding, forming, and perforating of the component have been completed.

3.4.4 Pre-plating stress relief and shot peening

3.4.4.1 All uncarburized steel parts having a hardness greater than Rockwell C40 shall be stress relieved prior to cleaning or plating by heating at 375°F ($\pm 25^\circ$) for 4 hours.

3.4.4.2 Unless otherwise specified by the engineering drawing, Class 2 plated steel parts shall be shot peened as follows:

- Plated parts below Rockwell C40 hardness and subjected to static loads or designed for limited life under dynamic loads or combinations thereof, shall not require shot peening prior to plating or baking after plating.

- Plated parts below Rockwell C40 hardness, which are designed for unlimited life under dynamic loads, shall be shot peened in accordance with Specification MIL-S-13165 prior to plating. Unless otherwise specified, the shot peening shall be accomplished on all surfaces for which the plating is required and on all immediately adjacent surfaces when they contain notches, fillets, or other abrupt changes of section size where stresses will be concentrated.

- Plated parts that have a hardness of Rockwell C40 or above, and are designed for unlimited life under dynamic loads, shall be shot peened in accordance with Specification MIL-S-13165 prior to plating. Unless otherwise specified, the shot peening shall be accomplished on all surfaces for which the coating is required and on all immediately adjacent surfaces when they contain notches, fillets, or other abrupt changes of section size where stresses will be concentrated.

3.4.5 Cleaning and conditioning basis metals

3.4.5.1 Steels of less than 7 percent alloying elements

A. Cleaning - Components made of these steels shall be cleaned in the manner outlined in Specification MS 101.3.

B. Etching - Immerse components in an aqueous solution containing from 16 to 60 ounces of chromic acid (CrO_3) per gallon of solution, at a temperature from 70 to 130°F and anodically etch for 30 to 90 seconds at a current density of 100 to 300 asf.

3.4.5.2 Corrosion or heat resistant steels

A. Cleaning - Components made of these steels shall be cleaned in the manner outlined in Specification MS 150.4. The electropolishing operation shall be optional.

B. Acid etch - A bath composed of 5 to 50 percent by volume hydrochloric acid at room temperature, an immersion time from 1 to 5 minutes, the work made cathodic, and a current density of 20 amperes per square foot shall be used.

C. Nickel strike - In a second tank containing a solution of the following analysis:

Nickel chloride	33-40 oz/gal
Hydrochloric acid	12-20 oz/gal
Iron	0.75 percent, max
Temperature	Room
Electrodes	Nickel

The work is made anodic for 2 minutes at 20 asf followed by cathodic treatment at 20 asf for 1 to 3 minutes.

D. Rinse - Rinsing in cold tap-water should be thorough, followed by rapid transfer to the chromium plating tank.

3.4.5.3 Aluminum and aluminum alloys

A. Clean and deoxidize - Aluminum and aluminum alloy components shall be cleaned and deoxidized in the manner specified in paragraph 3.4 of Specification MS 100.2A.

B. Conditioning - Aluminum and aluminum alloy surfaces shall be conditioned, for subsequent zinc immersion treatment, in the applicable manner specified below:

(1) A13, 319, 356, and 380 casting alloys - Dip at ambient temperature for 3 to 5 seconds in an aqueous solution containing 3 percent by volume of nitric acid (sp. gr. 1.42) plus 1 percent by volume of hydrofluoric acid (48 percent).

(2) All wrought alloys and cast alloys not covered in (1) - Immerse in an aqueous solution containing 15 percent by volume of sulphuric acid (66-degree Baume) for a period of 2 to 5 minutes at a temperature of 175 to 180°F.

(3) Cast alloys, wrought alloys not containing magnesium, and unidentified alloys - Immerse in an aqueous solution containing 70 ounces of commercial sodium hydroxide (76 percent Na_2O), 13 ounces of zinc oxide (technical grade), 0.13 ounce of ferric chloride (anhydrous), and 1.3 ounces of Rochelle salts per gallon of solution at a temperature of 70°F ($\pm 10^\circ$) for 30 to 60 seconds. Rinse the component by immersion in ambient temperature tap water for 1 to 2 minutes. Remove the zinc layer by dipping the component into an aqueous solution containing 50 percent by volume of nitric acid (sp. gr. 1.42) at ambient temperature for 1 to 2 minutes. Thoroughly remove all traces of acid by rinsing in clean, flowing tap water at ambient temperature for 1 to 2 minutes.

C. Zinc immersion coating - Immerse all components in an aqueous solution containing 70 ounces of commercial sodium hydroxide 76 percent (Na_2O), 13 ounces of zinc oxide (technical grade), 0.13 ounce of ferric chloride (anhydrous), and 1.3 ounces of Rochelle salts per gallon of solution at a temperature of 70°F ($\pm 10^\circ$) for 30 to 60 seconds.

D. Rinse - Double rinse all components by means of two combination "dip and spray" rinses using tap water at ambient temperature until all traces of excess zincate solution have been removed.

NOTE: Do not allow parts to dry between final rinsing and plating.

E. Plate - All aluminum components which have been cleaned and conditioned for plating as specified in paragraphs 3.4.5.3.A through 3.4.5.3.D shall be plated as specified in paragraph 3.4.6.

3.4.5.4 Copper and copper alloys - Components made of these materials shall be cleaned as follows:

A. Scale removal - When heavy scale, such as results from heat treatment, must be removed, parts shall be pickled in an aqueous solution of 5 to 10 percent by volume of sulfuric acid at room temperature for 1 to 15 minutes.

B. Degreasing - Parts shall be degreased per Specification MS 150.0.

C. Alkaline degrease - Parts shall be immersed in an aqueous solution of Wyandotte B. N., or equal, at a concentration of 4 ounces per gallon, at a temperature of 190 to 210°F and for 2 to 20 minutes depending on the degree of soil.

D. Rinse - Parts shall be rinsed by dipping in cold water.

E. Electroclean - Parts shall be electrocleaned in an aqueous solution of Wyandotte B. N. at a concentration of 4 to 6 ounces per gallon, at a temperature of 150 to 180°F, at a current density of 20 to 50 amperes per square foot for 1 to 3 minutes cathodically and for 5 to 10 seconds anodically.

F. Bright dip - To remove tarnish and stain as well as to smoothen the surface, parts shall be immersed in a bath composed of 20 percent by weight nitric acid, 25 percent by weight glacial acetic acid, 54.5 percent by weight phosphoric acid, and 0.5 percent hydrochloric acid. The bath shall be operated at 190°F ($\pm 5^\circ$) and the time of immersion may be up to 5 minutes.

G. First rinse - Parts shall be rinsed by immersion in water at 150°F ($\pm 10^\circ$).

H. Second rinse - Parts shall be immersed in a cold flowing rinse and transferred quickly to the plating bath.

3.4.6 Plating

3.4.6.1 Bath conditions - The following bath conditions, as noted in Table 4-13, are recommended for efficient and quality work:

A. The temperature of the bath should be held between 110 and 120°F. (See Note "a", Table 4-13).

B. The current density of the bath should be between 0.7 and 1.5 asi. (See Note "a", Table 4-13).

C. Agitation of the cathode is optional.

D. The cathode efficiency is approximately 13 percent.

E. The ratio of the anode-to-cathode area should be 1:1 to 2:1.

F. Voltage 6 to 12.

3.4.6.2 Immersion - Properly racked parts shall be immersed before applying the voltage. Parts shall remain in the bath for the required time before power is interrupted. If the coating thickness is inadequate, it shall be returned immediately to the bath to prevent layering of the second chromium deposit. If parts have been tarnished they shall be cathodically activated in a ten percent by weight hydrochloric acid solution for less than one minute. Parts shall not be immersed in the plating bath with live contacts to prevent uneven current distribution and possible burning.

3.4.6.3 Rinse - Parts shall be rinsed by immersion in hot water at 150°F ($\pm 10^\circ$) followed by a cold running rinse.

3.4.7 Post-plating hydrogen embrittlement relief - Within 1 hour after completion of plating, Class 2 plated parts having a hardness greater than Rockwell C40 shall be heated to 375°F ($\pm 25^\circ$) and maintained at temperature for 24 hours. No plated part of this hardness shall be flexed or stressed prior to baking and pursuant to baking it shall be tested in accordance with paragraph 4.3.4.

3.5 Appearance - Chromium plating shall be smooth, fine grained, adherent, free from visible blisters, pits, nodules, porosity, and indications of burning, excessive edge build up, and free from other detrimental defects. Slight discoloration resulting from baking as specified herein shall not be cause for rejection. All details of workmanship shall conform to the best practice for high-quality plating.

3.5.1 Class 1, Type I, plating shall be bright, smooth, uniform in appearance, and free from frosty areas.

3.5.2 Class 1, Type II, plating shall have a satin finish, smooth and uniform in appearance.

3.5.3 Class 2 plating on functional areas shall be smooth and free from frosty areas. Edges that cover only a portion of the surface shall, after finishing as indicated in the engineering drawing, be free of beads, nodules, jagged edges, and other detrimental irregularities.

3.6 Plating Thickness

3.6.1 Class 1 - Unless otherwise specified, the minimum plating thickness shall be as specified on all visible surfaces which can be touched by a ball 0.75 inch in diameter. The minimum thickness shall be 0.00001 inch.

3.6.1.1 Unless otherwise specified on the engineering drawing, Class 1 plating shall be applied over an intermediate coating of nickel or nickel plus copper, applied in the manner outlined in Specification MS 120.1. (See Table 4-15).

3.6.2 Class 2 - The minimum, maximum, or range of thickness of the chromium coating shall be as specified on the engineering drawing.

3.6.2.1 Class 2 plating is applied to parts for wear resistance and such incidental corrosion protection as the specified thickness of plate may afford. For additional corrosion protection, an undercoat of electrodeposited nickel, applied in the manner outlined in Specification MS 120.1, in thicknesses of 0.001 or 0.002 inch, should be used on ferrous parts. The minimum nickel thickness shall be determined by service conditions.

3.7 Control of Internal Stress - Plating shall be performed under operating conditions which also yield a control of the stress in the electrodeposited metal as measured by the Brenner-Senderoff contractometer or by Erichsen-Cup tests. The stress level shall not exceed that which will yield a satisfactory test result when samples or parts are tested in accordance with paragraph 4.3.4.

3.8 Adhesion - The adhesion of the chromium plating and any undercoat(s) shall be such that when examined at a magnification of approximately 4 diameters, neither the chromium plating nor any electrodeposited undercoat(s) shall show separation from the basis metal or from each other at their common

TABLE 4-15. NICKEL PLATINGS USED UNDER CHROMIUM PLATINGS
MINIMUM PLATING THICKNESSES (IN.)

Class of Chromium Plating	Plating	Plated Metal or Metals	Steel Basic Metal	Copper and Copper-Alloy Basic Metal	Aluminum and Aluminum Alloy Basic Metal								
Class 1*	Class 1	Cu Undercoat, in. Nickel, in. Ni ≠ cu, in. (1)	Optional 0.002 0.003	(2) 0.002 0.003	(3) 0.002 0.003								
						Type							
	Type			Type									
	Class 2*		DS	FS	KS	QS	FC	KC	QC	DA	FA	KA	QA
			0.001 0.002	0.0006 0.00125	0.0004 0.00075	0.0002 0.0004	(2) 0.0005	0.0003	0.0001	(3) .002	(3) .001	(3) .0006	(3) .0004 .00075

- * (1) Class 2 nickel platings, including copper undercoat, are ordinarily used as undercoat systems for Class 1 electrodeposited chromium.
- (2) Copper alloys containing zinc equal to or greater than 40 percent shall have a copper undercoat of 0.0004-inch minimum thickness for Class 1 plating and 0.0003-inch for Class 2 plating.
- (3) Aluminum alloys having a zinc immersion coating shall have a copper undercoat of 0.00065-inch minimum thickness for Class 1 nickel plating and 0.00035-inch for Class 2 nickel plating.

interface(s) when subjected to the test specified in 4.3.2. The interface between a plating and the basis metal is the surface of the basis metal before plating. The formation of cracks in the basis metal or plate which do not result in flaking, peeling, or blistering of the plate shall not be considered as nonconformance to this requirement.

4. QUALITY ASSURANCE PROVISIONS

4.1 General - To effectuate adequate control over the quality of plating, analyses shall be made of the composition of the plating bath and other processing baths at least once each month. Verification of analyses and tests, in the form of recorded data, shall be maintained. The results of all routine tests for stress, adhesion, thickness, and corrosion shall also be recorded.

4.2 Sampling

4.2.1 Lot - For the purposes of this specification, a lot is defined as a group of plated articles of the same size, the same shape, having the same type and class of chromium plating, and submitted for inspection at the same time.

4.2.2 Sampling for visual inspection and for non-destructive tests of plating thickness - A sample shall be drawn from each inspection lot by taking at random from the lot the percentage of articles specified in Table 4-16. These articles shall then be subjected to both the visual inspection and the plating thickness determination outlined in paragraphs 4.2.2.1 and 4.2.2.2, respectively.

4.2.2.1 Visual examination - All sample articles selected in paragraph 4.2.2 shall be visually examined to assure conformance to the requirements of paragraph 3.5. If the number of nonconforming articles exceeds the acceptance number for that size sample (see Table 4-16), the lot represented by that sample shall be rejected.

4.2.2.2 Thickness determination - All sample articles selected in paragraph 4.2.2 shall have their plating thickness measured by a non-destructive method at several locations on each article where the plating is expected to be of minimum thickness. An article shall be considered to be nonconforming if any measurement fails to meet the specified minimum thickness. If the number of nonconforming articles in any sample exceeds the acceptance number for that size sample (see Table 4-16), the lot represented by that sample shall be rejected.

TABLE 4-16. SAMPLING FOR NON-DESTRUCTIVE TESTS

Items in Inspection Lot (Number)	Items in Sample (Percent of Inspection Lot)		Acceptance Number (Maximum Allowable Number of Items Nonconforming)
	Visual , 4.2.2.1	Thickness, 4.2.2.2	
1 to 6	100	100	0
7 to 15	100	100	0
16 to 40	100	100	0
41 to 110	100	60	0
111 to 300	60	40	1
301 to 500	30	20	1
501 and over	20	15	2

TABLE 4-17. SAMPLING FOR DESTRUCTIVE TESTS

Items in Inspection Lot (Number)	Items In Sample (Number)	Acceptance Number (Maximum Allowable Number of Items Nonconforming)
1	0	0
2 to 5	1	0
6 to 25	3	0
26 to 100	4	0
101 to 300	6	0
301 to 5000	10	0
5001 and over	15	0

4.2.3 Sampling for destructive tests of plating thickness, adhesion, salt spray resistance, and hydrogen embrittlement relief - A random sample, of the size specified in Table 4-17, but composed of different articles than those selected for non-destructive tests in paragraph 4.2.2, shall be selected from each inspection lot. In addition, eight separate plated test specimens shall be prepared at the same time as production articles to represent each inspection lot.

4.2.3.1 Thickness tests - Each article of the sample selected in paragraph 4.2.3 shall be tested in several locations at which the plating would be expected to be a minimum. If the plating thickness at any location on any article is less than the specified thickness, the lot represented by that article shall be rejected.

4.2.3.2 Adhesion - All the articles used for the plating thickness tests, if of suitable size and form, shall be used as specimens for the adhesion tests. If the articles used for plating thickness tests are not of suitable size and form, an equal number of additional articles shall be selected or the four remaining separately plated test specimens used. Failure of any test specimen shall result in the rejection of the lot represented by that test specimen (reference paragraph 4.3.2).

4.2.3.3 Salt spray resistance - For Class 1 chromium plating, four of the eight separately plated test specimens prepared in paragraph 4.2.3 shall be tested in lieu of plated articles which comprise the inspection lot. The failure of any test specimen in less than the time specified in Table 4-18, for the type nickel-copper undercoat system used, shall result in rejection of the lot represented by that test specimen (reference paragraph 4.3.3).

4.2.3.4 Hydrogen embrittlement relief - A random sample of the size specified in Table 4-17 shall be selected from each lot of plated articles and tested for hydrogen embrittlement relief in the manner specified in paragraph 4.3.4. Failure of any of the articles tested shall result in the rejection of the lot represented by that article.

4.3 Test Methods

4.3.1 Thickness - Thickness determinations shall be made by the microscope method, or an instrument or chemical method approved by the MSFC initiating activity. For referee tests, the microscopic method shall be used. Measurement of minimum plating thickness on bolts shall be made on the shank adjacent to the thread, or on the head if the shank is completely threaded.

TABLE 4-18. SALT SPRAY EXPOSURE LIFE

Basic Metal	Type Nickel - Copper Undercoat	Minimum Time To Failure (hr)
Steel	DS	96
	FS	72
	KS	48
	QS	16
Copper and Copper Alloys	FC	72
	KC	48
	QC	16
Aluminum and Aluminum Alloys	DA	192
	FA	120
	KA	72
	QA	32

4.3.1.1 Microscopic method - A suitable procedure for the microscopic determination of the thickness of the chromium plating is as follows: A test specimen consists of a cross-section of a selected surface. One or more specimens are mounted in a plastic material such as a phenolic or acrylate resin. The specimen must be held rigidly and in such a position that the surface to be tested is perpendicular to the flat face that is to be ground and polished. (A deviation of 10 degrees from normal introduces an error of about 2 percent in thickness.) The specimen may be mounted at a known angle to obtain a greater effective magnification and the thickness measured then corrected for the angle. The mounted specimen is ground and polished using successively finer metallographic polishing papers, the last of which should not be less than 500 mesh. The polished surface is then etched with a suitable solution such as the following:

Concentrated nitric acid (sp. gr. 1.42) 2 ml.
Ethyl alcohol 95 percent 100 ml.

The thickness may be measured with an accurately calibrated filar ocular micrometer, or the image may be measured with an accurate ruler on a ground-glass focusing plane of a metallographic microscope.

4.3.2 Chemical Method

4.3.2.1 Reagent - Reagent grade hydrochloric acid having a specific gravity of $.180 \pm 0.002$ at 60/60°F. shall be used. (This corresponds to 11.5N ± 0.2 N, which may be checked by titration.)

4.3.2.2 Procedure - At each spot to be tested, the surface shall be cleaned by light rubbing with a paste of pure magnesium oxide and water, followed by rinsing and drying. At each spot, an area about 0.25 inch (0.6 cm) in diameter shall be surrounded by a small ring of wax. (A wax pencil may be used.) The article shall then be allowed to stand long enough (at least 15 minutes) to reach the room temperature, which shall be between 65 and 85°F (18 and 30°C) and shall be noted within 2°F or 1°C. The acid shall also be kept in the room until it acquires room temperature. One drop (0.03 to 0.05 ml) of the hydrochloric acid shall then be placed within the wax ring. The time from the beginning of gas evolution until the gas evolution practically ceases and reveals the exposed nickel shall be determined to the nearest 1/2 second with a stop watch. If the bubbling does not start at once, the surface of the chromium within the ring may be touched with a fine nickel wire to start the reaction.

4.3.2.3 Thickness - The thickness of the chromium at the spot tested shall be calculated from the curve in Figure 4-23 by multiplying the factor for the temperature used in the test by the number of seconds required. For example, if 16 seconds are required at 72° F (22° C), reference to Figure 4-23 b gives a factor for this temperature of 0.086 hundred-thousandths of an inch per second. The thickness is therefore equal to $16 \times 0.086 = 1.4$ hundred-thousandths inch or 0.00014 inch. To meet the specified minimum thickness of 0.00010 inch of chromium at any definite temperature, the periods designated in Table 4-19a will be required.

4.3.2.4 Instrument method - Suitable magnetic or eddy current measuring instruments (Magne-Gage produced by American Instrument Co., Inc., 8030 Georgia Ave., Silver Spring, Md., or equal) may be used for measuring the plating thickness on steels in lieu of the microscopic method.

4.3.3 Adhesion

4.3.3.1 Scrape method - Adhesion may be determined by scraping the surface of the plated article, or test specimen, to expose the basis metal. The junction of the plating and the exposed basis metal shall then be examined at four diameters magnification. Any evidence of flaking, blistering, or separation between the plating and the basis metal shall be interpreted as unsatisfactory adhesion.

4.3.3.2 Bend method - Adhesion may be determined by clamping the plated article, or test specimen, in a suitable holding device and rapidly bending at room temperature through an angle of 180 degrees, around a diameter equal to the thickness of the specimen, until plating rupture occurs. If the edge of the ruptured plating can be peeled back or if separation between the plating and the basis metal can be seen at the point of rupture when examined at four diameters magnification, adhesion shall be interpreted as unsatisfactory.

4.3.4 Salt spray resistance - Salt-spray resistance test shall be made in the manner specified in Method 811.1 of Federal Test Method Standard Number 151a.

4.3.5 Hydrogen embrittlement relief - Hydrogen embrittlement relief tests shall be made by subjecting each of the plated articles in the lot sample to the maximum stress to which it is intended that the articles be exposed in actual service. This stress shall be specified by the MSFC Procuring Activity and shall be constantly maintained for a minimum period of 240 hours. The failure of any test article in less than 240 hours shall be interpreted as unsatisfactory hydrogen embrittlement relief of the lot represented by that article.

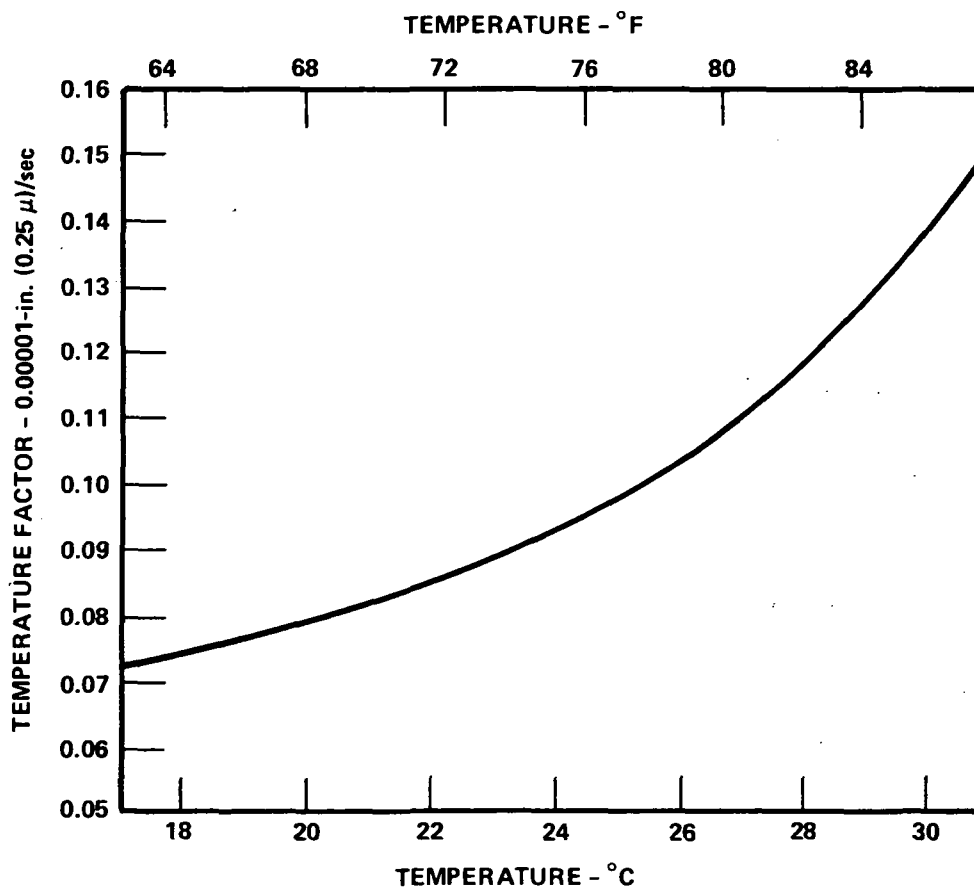


Figure 4-23 b. Temperature factor for hydrochloric acid spot test for thickness of electrodeposited chromium; specific gravity of HCl, 1.180.

TABLE 4-19a. PENETRATION TIME FOR 0.000010-INCH
ELECTRODEPOSITED CHROMIUM

Test Temperature in Degrees		Penetration Time (Seconds)
Fahrenheit	Centigrade	
64	17.8	13.5
66	18.9	13
68	20	12.5
70	21.1	12
72	22.2	11.5
74	23.4	11
76	24.5	10.5
78	25.5	10
80	26.7	9.5
82	27.8	8.5
84	28.9	8.
86	30	7.5

4.4 Retests - Plated articles which have been subjected to a chemical method of plating thickness determination and articles rejected or withdrawn due to defective or deficient plating may be resubmitted after stripping and replating of the individual articles in the first case and of the entire lot in the second case.

4.5 Analysis of Plating Solutions

4.5.1 Determination of chromic acid - Proceed as follows:

- Pipette a 10-ml sample into a 500-ml volumetric flask, dilute to the mark with distilled water and mix thoroughly.
- Pipette 10 ml of the solution prepared under the above step into a 250-ml Erlenmeyer flask.
- Add 100 ml of distilled water.
- Add approximately 2 g of ammonium bifluoride.
- Add 15 ml of concentrated hydrochloric acid.
- Add 10 ml of 10-percent potassium iodide.
- Fill the 50-ml burette with standard sodium thiosulfate solution and titrate until the dark-brown color begins to fade.
- Add 5 ml of a 1-percent starch indicator solution and continue titrating until the blue color disappears for about 1 minute.
- Read the burette. This value multiplied by the proper factor for standard sodium thiosulfate solution gives the ounces of chromic acid per gallon of the plating bath.

4.5.1.1 Preparation of standard sodium thiosulphate solution

- Weigh 24.82 grams of reagent-grade sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3 - 5\text{H}_2\text{O}$) and dissolve in distilled water.
- Add 0.1 gram of sodium carbonate then dilute to 1 liter with distilled water in a volumetric flask.

- Standardize the sodium thiosulphate solution against reagent potassium dichromate as outlined in the following steps.
- Weigh 0.1 gram of pulverized potassium dichromate which has been previously dried for 2 hours at 230° F.
- Transfer the potassium dichromate to a 240 ml-Erlenmeyer flask, add 100 to 125 ml of distilled water and 2 grams of ammonium bifluoride.
- Add 15 ml of concentrated hydrochloric acid and 10 ml of a 10 percent potassium iodide solution.
- Fill a 50-ml burette with the standard sodium thiosulphate solution and titrate until the dark-brown color begins to fade.
- Add 5 ml of a 1 percent starch indicator solution and continue titrating until the blue color disappears for 1 minute.
- Read the burette.
- Calculate the factor as follows:

$$\frac{\text{Weight of K}_2\text{Cr}_2\text{O}_7 \times 0.679 \times 5000}{\text{Milliliters of standard Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O} \times 7.5} = \text{Factor in oz/gal of chromic acid (CrO}_3\text{)}.$$

4.5.2 Determination of sulphate - Proceed as follows:

- Use a centrifuge for the sulfate determination.

CAUTION: Be careful in the use of this apparatus. Always start the centrifuge slowly. Keep away from the revolving tubes.

- Add 5 ml of 75-percent by volume hydrochloric acid to one centrifuge tube.
- Pipette a 20-ml sample into the same tube, stopper and mix.
- If the solution is warm, allow to cool.
- Add 5 ml of 25-percent barium chloride and shake vigorously for 1 minute.

- Place in a centrifuge holder and allow to stand for 5 minutes.
- Add 30 ml of distilled water to the other centrifuge tube, stopper and place in the other holder.
- Turn the handle of the centrifuge for 1/2 minute at a rate of about 60 rpm.
- Remove the sample tube and tap the stem with the finger until the precipitate has a flat surface.
- Replace sample tube in holder and centrifuge again for about 15 seconds.
- Remove the tube and read the volume of the precipitate in the stem. Each marked division equals 0.1 oz/gal of sulfate; each smallest division is 0.02 oz/gal of sulfate.

NOTE: All operations must be timed exactly.

If the precipitate is above the 0.4 oz/gal mark, repeat the test using a 10-ml sample in the 3rd step, and 20 ml of water instead of 30 ml in the 7th step. After making the final reading, multiply the result by 2 to obtain the correct concentration of sulfate in oz/gal, i.e., 0.28 on the stem will equal 0.56 oz/gal of sulfate.

4.5.3 Determination of trivalent chromium - Proceed as follows:

- Pipette a 10-ml sample into a 500-ml volumetric flask. Dilute to the mark with distilled water and mix thoroughly.
- Pipette a 10-ml aliquot into a 250-ml Erlenmeyer flask and add 100 ml of distilled water.
- Add 0.2 g of sodium peroxide (Na_2O_2).
- Boil at least 20 minutes but not exceeding 30 minutes.
- Dilute to between 100 and 125 ml and allow to cool.
- Add 2 g of ammonium bifluoride (NH_4HF_2).

- Add 15 ml of concentrated hydrochloric acid and allow to cool.
- Add 10 ml of potassium iodide solution (100 g/l KI \pm 1 g/l KOH).
- Fill a 50-ml burette with standard sodium thiosulfate solution and titrate until the brown color changes to a straw color.
- Then add 3 ml of a starch solution (10 g/l soluble starch).
- Continue titration until the blue color just disappears.
- Read the burette. This value multiplied by the factor listed below for the standard sodium thiosulfate solution ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) gives the total chromium as equivalent chromic acid (CrO_3). To calculate the trivalent chromium proceed as follows:

(oz/gal total chromium as CrO_3) — (oz/gal of actual CrO_3) \times 0.52 =
oz/gal trivalent chromium ($\text{Cr} \pm 3$) in the plating bath.

4.5.3.1 Preparation of standard sodium thiosulphate solution (See paragraph 4.5.1.1).

4.5.4 Determination of iron - Proceed as follows:

- Pipette a 10-ml sample into a 250-ml beaker and dilute to 50 ml with distilled water.
- Add a 40-percent NaOH solution until a precipitate begins to form.
- Cover the beaker with a watch glass, add 1 g of Na_2O_2 then 25 ml of bromine water and boil for 15 minutes.
- Dilute to 90 ml, washing the beaker and watch glass into the solution.
- Filter through No. 1 Whatman filter paper (or equivalent) discarding filtrate.
- Wash at least 10 times with hot water.

- Dissolve the precipitate in the original beaker with hot 50-percent HCl and wash the paper until free from acid.
- If the solution is green in color (do not mistake it for the yellowish color of the ferric chloride complex), repeat steps 2 through 8.
- Add concentrated ammonium hydroxide (sp. gr. 0.897) until a precipitate forms, add an excess of 2 ml, then heat to boiling.
- Filter through No. 1 Whatman filter paper into a 250-ml Erlenmeyer flask and wash the beaker and precipitate with 25-percent NH_4OH and then 5 times with warm water.
- Dissolve the precipitate into the original beaker with hot 20-percent sulfuric acid, washing the filter paper 5 times with hot water.
- Add 25 ml of 50-percent sulfuric acid and dilute to between 100 and 150 ml.
- Place an aluminum wire coil in the beaker, boil for 30 minutes and remove the coil, washing it off in the beaker with water.
- Fill a 50-ml burette with standard potassium permanganate (KMnO_4) solution and titrate until the solution changes to a pale-pink color permanently for 1 minute.
- Read the burette. This value multiplied by the factor for the standard potassium permanganate solution gives the ounces of iron (Fe) per gallon of plating bath.

4.5.4.1 Preparation of standard potassium permanganate solution

- Dissolve 3.3 grams of potassium permanganate (KMnO_4) in distilled water, fill up to a liter, and allow to stand for at least 1 week.
- Filter the solution through a fritted glass filtering funnel and standardize as in the following steps.

- Accurately weigh out 0.25 gram of dried analytical reagent sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$).
- Transfer all the $\text{Na}_2\text{C}_2\text{O}_4$ to a 500 ml Erlenmeyer flask and add 150 ml of distilled water.
- Add 15 ml of 20 percent H_2SO_4 and heat to 160 to 175°F.
- Fill a 50 ml burette with the potassium permanganate solution and titrate the hot sodium oxalate solution until it changes to a pale permanent pink color for 1 minute.
- Read the burette and use the value obtained to calculate the iron factor as follows:

$$\frac{\text{Weight of } \text{Na}_2\text{C}_2\text{O}_4 \times 0.833 \times 100}{\text{Milliliters of standard } \text{KMnO}_4 \times 7.5} = \text{Factor in oz/gal for iron (Fe)}.$$

5. PREPARATION FOR DELIVERY

Not applicable to this specification.

6. NOTES

6.1 Intended Use - This manufacturing specification, developed by the PE Laboratory of George C. Marshall Space Flight Center, is intended for use when chromium plating is performed on space vehicle components.

6.2 Important Considerations

6.2.1 Ammonium salts - Ammonia and ammonium salts must be kept out of chromium baths. They cannot be removed.

6.2.2 Grease, oil, and buffing compound - See that such materials are removed in pretreatment processes, either by use of the over-flowing tank feature or by removing them from the surface of the bath by blotting with clean newspapers.

6.2.3 Fallen parts - Do not allow parts to accumulate in the bottom of the tank. They will dissolve and contaminate the bath.

6.2.4 Copper flash - Take care that the copper plating and nickel plating solutions used prior to chromium plating are kept in good condition. Rough copper deposits result in rough nickel coatings, rough nickel coatings result in rough chromium platings. Also rinse very thoroughly after each particular plating operation to avoid copper or nickel contamination of the chromium bath.

NOTICE - When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING SPECIFICATION FOR ELECTROLESS NICKEL PLATING

1. SCOPE

1.1 Scope - This manufacturing specification covers the approved PE Laboratory methods and requirements for electroless nickel plating of components to be used in space launch vehicle and support equipment applications.

1.2 Applicability - The methods and requirements set forth in this specification are applicable to electroless nickel plating of the following wrought, cast, or forged metals and alloys:

- Magnesium and magnesium Alloys
- Aluminum and aluminum alloys
- Iron and iron-base alloys
- Nickel and nickel alloys
- Copper and copper alloys

1.3 Limitations - The methods and requirements set forth in this specification are not suitable for use in electroless nickel plating directly on the following metals or on surfaces contaminated by these metals.

- Lead
- Tin
- Zinc
- Cadmium
- Antimony
- Arsenic
- Bismuth
- Manganese

When it is desired to electroless nickel plate surfaces made of or contaminated by such metals, the surfaces should first be given a copper flash by the electrolytic method then plated with electroless nickel as though they were copper.

1.4 Classification - Electroless nickel plating shall be of the following classes and types:

Class 1 - Engineering plating intended for improvement of corrosion resistance, wear or abrasion properties and plated to specified dimensions or processed to specified dimensions after plating.

Type I	0.002 inch Ni, minimum.
Type II	0.001 inch Ni, minimum (Minimum for Fe or Al-based alloys unless otherwise specified on drawing).
Type III	0.00075 inch Ni, minimum.
Type IV	0.00050 inch Ni, minimum (Minimum for Cu, Ni, or Co-based alloys unless otherwise specified on drawing).
Type V	0.00025 inch Ni, minimum.
Type VI	0.0001 inch Ni, minimum.

Class 2 - Decorative plating

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this specification, form a part of this specification to the extent referenced herein.

SPECIFICATIONS

Federal

O-C-275	Ammonium Chloride
O-C-275	Ammonium Hydroxide

O-C-275	Ammonium Sulphate
O-C-275	Boric Acid
O-C-275	Hydrochloric Acid
O-C-275	Potassium Dichromate
O-C-275	Sodium Acetate
O-C-275	Sodium Carbonate
O-C-275	Sodium Citrate
O-C-275	Sodium Hydroxide
O-C-275	Sulphuric Acid
O-G-491A	Glycerin

Military

JAN-A-183(2)	Nitric Acid
MIL-N-21967 (Ships)	Nickel Salts, Electroplating
MIL-C-26074A	Coating, Nickel-Phosphorus, Electroless Nickel, Requirements for

George C. Marshall Space Flight Center

MS 100. 2A	Manufacturing Specification for the Deoxidation of Aluminum Alloys
MS 100. 4A	Manufacturing Specification for the Cleaning and Passivation of Austenitic (300 series) Stainless Steel Suction Lines
MS 100. 9	Manufacturing Specifications for the Cleaning of Miscellaneous Components

MS 101.3	Manufacturing Specification for the Cleaning of Carbon Steel Alloys
MS 120.0	Manufacturing Specification for Cadmium Plating of Aluminum, Copper, and Steel Alloys
MS 150.0	Manufacturing Specification for Vapor Degreasing of Metallic Surfaces
MS 150.1	Manufacturing Specification for Sand and Vapor Blasting
MS 150.2	Manufacturing Specification for Anodizing of Aluminum Alloys
MS 150.4	Manufacturing Specification for Electro-polishing of Corrosion Resistant Steel

STANDARDS

Military

MS 36051	Selenic Acid
MS 36133	Palladium Chloride

2.2 Other Publications - The following publications form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

Society of Automotive Engineers, Inc.

AMS 2404	Electroless Nickel Plating
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(Copies of the publication listed above may be obtained from the Society of Automotive Engineers, Inc., 485 Lexington Ave., New York 17, N. Y.)

3. REQUIREMENTS

3.1 General - No deviation from the requirements of this specification shall be permitted without prior written approval of the initiating activity.

3.2 Base Metal Forming Operations - Unless otherwise specified, plating shall be applied only after all machining, brazing, welding, forming, and perforating of the component have been completed.

3.3 Equipment

3.3.1 Cleaning and plating tanks - Tanks of a suitable size, constructed of or lined with glass, plastic-coated or glass-lined carbon steel, stainless steel (bare 400 series or Type 304), enameled steel, or glazed stoneware shall be used for containing electroless nickel plating solutions.

3.3.2 Part supports - Bars, hangers, racks and jigs used for holding parts in position in solutions during electroless nickel plating shall be fabricated from one of the following materials:

- Steel, covered by a heavy coat of soft rubber, neoprene, or a thick temperature-and-acid-resistant plastic film such as vinyl plastisol.
- Heavily anodized aluminum
- Bare, passivated stainless steel, Type 304 or 400 series
- Silica or cobalt glass

3.3.3 Other system tanks and components - Make-up tanks, regeneration vessels, etching tanks, sensitizing tanks, activation tanks, and piping intended for use at temperatures below 140 °F may be constructed of carbon steel lined with a baked phenolic coating or from bare 400 series or Type 304 stainless steel.

3.4 Materials

3.4.1 Chemicals - The chemicals used in the preparation of electroless nickel processing solutions shall be C. P. (chemically pure) grade or highest purity technical grade.

3.4.2 Water - The water used in the preparation of electroless nickel processing solutions shall be distilled or demineralized water having specific resistance of 50 000 ohms.

3.4.3 Masking tape - Masking tape shall be Electroplaters' Tape No. 470 (Minnesota Mining and Manufacturing Co.), or equal.

3.5 Preplating Operations

3.5.1 Preparation of plating solutions

3.5.1.1 Prefilling tanks - Tanks in which plating solutions are to be mixed shall be filled approximately three-fourths full with distilled or demineralized water heated to 175°F ($\pm 5^\circ$).

3.5.1.2 Chemical additions - Using constant agitation, slowly add the sodium hypophosphite, then buffering or chelating agents, then the nickel salt which has previously been dissolved in hot water.

3.5.1.3 pH adjustment - Adjust the pH (hydrogen ion concentration) of the tank to the value specified in Table by slowly adding the diluted alkali solution specified in Table 4-19b.

3.5.1.4 Final filling of tanks - Fill tanks to the final desired volume by adding room temperature distilled or demineralized water.

3.5.1.5 Filtering - Allow the freshly mixed plating solution to stand at least overnight then filter to remove all solid particles.

3.5.2 Plating solution analyses - After final filtering, typical electroless nickel plating solution analyses shall be as specified in Table 4-19 as applicable.

3.5.3 Cleaning of Components

3.5.3.1 General - All surfaces to be plated shall be thoroughly cleaned prior to the application of the nickel plate. Meticulous cleaning and surface preparation of the components cannot be overemphasized since this factor is of prime importance in obtaining a satisfactorily adhering plating.

3.5.3.2 Magnesium and magnesium alloys - Magnesium and magnesium alloys shall be thoroughly degreased in the manner outlined in Specification MS 150.0 followed by immersion for 3 to 10 minutes at 190 to 212°F in an alkaline cleaning bath containing 3 ounces of sodium carbonate ($\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$) and 2 ounces of sodium hydroxide (NaOH) per gallon of solution. Parts may be cathodically cleaned in the alkaline cleaner at a current density of 10 to 40 amperes per square foot at approximately 6 volts. After cleaning, parts shall be thoroughly rinsed by immersion in running water at room temperature.

**TABLE 4-19b. ELECTROLESS NICKEL BATH COMPOSITIONS
AND OPERATING CONDITIONS**

INGREDIENTS	Type 1 (Acid)	Type 2 (Alkaline)	Type 3 (Acid)				FUNCTION
$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$, oz/gal	4.0-4.5	4.5-6.0	—				Supply Nickel
$\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, oz/gal	—	—	4.9-5.1				Supply Nickel
Sodium compounds, oz/ gal							
Hypophosphite	1.33-1.5	1.47	1.9-2.1				Reducing agent
Hydroxy acetate	1.33-7.0	—	—				Buffer and chelating agent
Citrate	—	13.3-14.0	0.6				Buffer and chelating agent
Acetate	—	—	0.5				Buffer
NH_4Cl , oz/gal	—	6.67-7.0	—				Buffer and complexing agent
Acids							
Boric, oz/gal	—	—	—				
Lactic, 80 percent oz/gal	—	—	—				
Selenic, ppm	0.1-10.0	—	—				Buffer, chelating agent and exaltant
Propionic, fl. oz/gal	—	—	—				
Lead, ppm	—	—	—				Stabilizer
Tellurium, ppm	—	—	—				Stabilizer and brightener
Conditions							
pH, electrometric	4-6	8.0-10.0	4.0-6.0				
Adjust pH using	NaOH	NH_4OH	NH_4OH				
Temperature, F	190±2	194-212	185-200				
Plating rate, Mil/hr	0.6	0.3	.35				
Coating appearance (1)	SB	B	SB				

(1) SB-Semi-bright, B-Bright, S-Smooth, VB-Very Bright

3.5.3.3 Zinc-base or high-zinc alloys - Zinc-base or high-zinc alloys shall be thoroughly degreased in the manner outlined in Specification MS 150.0 followed by anodic cleaning for 30 to 90 seconds at 150 to 190 °F in an alkaline cleaning bath containing 5 to 80 ounces of Oakite Composition No. 195, or equal, per gallon of solution. Current density should be from 40 to 50 amperes per square foot with the current being applied only after work is completely immersed in the solution. After anodic cleaning, parts shall be thoroughly rinsed by immersion in running water at room temperature.

3.5.3.4 Aluminum and its alloys - Aluminum and its alloys shall be cleaned in the manner outlined in Specification MS 100.2A.

3.5.3.5 Iron-base alloys

A. Low-carbon steels - Low carbon steels shall be cleaned in the manner outlined in paragraphs 3.4.4.3.3 through 3.4.4.3.9 of Specification MS 120.0.

B. High carbon and tool steels - High carbon steels shall be cleaned in the manner outlined in paragraphs 3.4.4.2.A through 3.4.4.2.O of Specification MS 120.0.

C. Stainless steels - Stainless steels shall be cleaned in the manner outlined in paragraphs 3.4.4.4.A through 3.4.4.4.G of Specification MS 120.0.

3.5.3.6 Cobalt and cobalt-base alloys - Cobalt and cobalt-base alloys shall be thoroughly degreased in the manner outlined in Specification MS 150.0 followed by immersion for 1 to 2 minutes at 70 to 80 °F in an aqueous solution containing 20 (±1) percent by volume hydrochloric acid (sp. gr. 1.098). Thoroughly rinse by immersion in running water at room temperature.

3.5.3.7 Nickel and nickel-base alloys - Nickel and nickel-base alloys shall be thoroughly degreased in the manner outlined in Specification MS 150.0 followed by immersion for 2 to 3 minutes at 180 to 190 °F in an alkaline cleaning bath containing 3 ounces of sodium carbonate and 2 ounces of sodium hydroxide (NaOH) per gallon of solution. Thoroughly rinse by immersion in running water at room temperature.

3.5.3.8 Copper, brasses and bronzes - Copper, brass, and bronze shall be cleaned in a manner outlined in paragraphs 3.4.4.5.A through 3.4.4.5.G of Specification MS 120.0.

3.5.4 Pickling - Immediately following the cleaning steps outlined in 3.5.3, metals and alloys shall be processed through the acid or alkaline baths in the manner outlined below.

3.5.4.1 Magnesium and magnesium alloys - Magnesium and magnesium alloys shall be successively processed through three different pickling baths immediately following the cleaning outlined in paragraph 3.5.3.2. The compositions, operating conditions, and sequence of processing through the baths shall be shown in Table 4-20.

3.5.4.2 Zinc-base or high-zinc alloys - Zinc-base or high zinc alloys shall be dipped for 15 to 30 seconds into an aqueous solution containing 1/4 to 1 percent sulphuric acid (96 percent) by volume, or 1/4 to 1 percent hydrochloric acid (sp. gr. 1.098) by volume, or 1/4 to 1 percent hydrofluoric acid (70 percent) by volume at room temperature. Upon removal from the acid solution, rinse thoroughly in demineralized water.

3.5.4.3 Aluminum and its alloys

(a) A 13, 319, 356, and 380 casting alloys shall be dipped at ambient temperature for 3 to 5 seconds in an aqueous solution containing 3 percent nitric acid (sp. gr. 1.42) by volume and 1 percent hydrofluoric acid (48 percent) by volume followed by thorough rinsing by immersion in clean tap water at ambient temperature for 1 to 2 minutes.

(b) Cast or wrought alloys not containing magnesium, and unidentified alloys shall be immersed in an aqueous solution containing 70 ounces of commercial sodium hydroxide (76 percent Na_2O), 13 ounces of zinc oxide (technical grade), 0.13 ounce of ferric chloride (anhydrous), and 1.3 ounces of Rochelle salts per gallon of solution at a temperature of 70°F ($\pm 10^\circ$) for 30 to 60 seconds. Rinse by immersion in tap water at ambient temperature for 1 to 2 minutes. Remove the zinc layer by dipping the component into an aqueous solution containing 50 (± 5) percent nitric acid (sp. gr. 1.42) by volume at ambient temperature for 1 to 2 minutes. Thoroughly rinse by immersion in clean tap water at ambient temperature for 1 to 2 minutes.

(c) All wrought or cast alloys not covered by (a) or (b) above shall be immersed in an aqueous solution containing 15 (± 1) percent sulphuric acid (66° Baume) by volume for 2 to 5 minutes at 175 to 180 °F. Thoroughly rinse by immersion in clean tap water at ambient temperature for 1 to 2 minutes.

TABLE 4-20. MAGNESIUM PICKLING BATHS

Bath Constituents and Operating Conditions	Bath Number and Order of Use					
	1			2		3
APPLICATION	A	B	C	D	E	F
Chromic acid, g/l	120	60	-			
Nitric Acid, ml/l	110	90	-			
Phosphoric acid, (85 percent), ml/l	-	-	900			
Hydrofluoric acid (70 percent), ml/l	-	-	-	54	220	
Tetrasodiumpyrophosphate, g/l	-	-	-	-	-	65
Time, minutes	1/2-2	1/2-2	1/2-2	10	10	3
Temperature, °F	75	75	75	75	75	150

A - General purpose, prior to direct electroless nickel.

B - Aluminum-free alloys, prior to direct electroless nickel.

C - High-aluminum alloys, prior to direct electroless nickel.

D - Alloys containing less than 5 percent aluminum.

E - Alloys containing more than 5 percent aluminum.

F - All magnesium alloys

*Budinkas, P., U. S. Patent No. 2,983,634 (May 9, 1961)

3.5.4.4 Iron-base Alloys

A. Iron and low carbon steels - Immerse in an aqueous solution containing 4 to 10 percent sulphuric acid (66° Baume) by volume at ambient temperature for 5 to 15 seconds. Immediately rinse by immersion in clean tap water at ambient temperature for 10 to 15 seconds. In addition, parts shall be subjected to a spray of clean tap water as they leave the rinse tank.

B. High carbon and tool steels - Immerse in an aqueous solution containing 3 to 4 ounces of sodium cyanide (NaCN) per gallon of solution at 70 to 80 °F for 15 to 30 seconds. Rinse thoroughly by immersion in or spraying with demineralized water.

C. Stainless steels - Immerse in an aqueous solution containing from 5 to 50 percent sulphuric acid (66° Baume) by volume at a temperature of 70°F ($\pm 5^\circ$) for 1 to 5 minutes. Subject all surfaces to a cathodic current density of 5 to 10 amperes per square foot for the entire 1 to 5 minutes. Rinse thoroughly by immersion in demineralized water at room temperature.

3.5.4.5 Cobalt and cobalt-base alloys - Immerse in an aqueous solution containing 67 ($\pm 5^\circ$) for 1/2 to 1 minute. Rinse thoroughly by immersion in or by spraying with demineralized water at ambient temperature for 1 to 2 minutes.

3.5.4.6 Nickel and nickel-base alloys

(a) Immerse nickel and "Z" nickel in an aqueous solution containing 31 (± 5) percent sulphuric acid (66° Baume) by volume, 47 (± 5) percent nitric acid (38° Baume), and 1 (± 0.15) ounce of sodium chloride per gallon of solution at a temperature of 70 to 100° F for 5 to 20 seconds. Thoroughly rinse by immersion in room temperature tap water, then neutralize by dipping into a room temperature aqueous solution containing 1 to 2 percent ammonia by volume followed by a thorough final rinse in demineralized water at room temperature.

(b) Immerse Monel, "K" Monel, and "R" Monel in an aqueous solution containing 50 (± 5) percent nitric acid (38° Baume) by volume and 4 to 6 ounces of sodium chloride per gallon of solution at a temperature of 70 to 100° F for 4 to 6 seconds. Rinse by immersion in tap water at 180 °F ($\pm 10^\circ$) then immerse in a second aqueous acid bath containing 50 (± 5) percent nitric acid (38° Baume) by volume at a temperature of 70 to 100 ° F for not more than 5 seconds. Rinse again by immersion in tap water at room temperature then neutralize by dipping into a room temperature aqueous solution containing 1 to 2 percent commercial ammonia by volume, followed by a thorough final rinse in demineralized water at room temperature.

(c) Immerse Inconel, Inconel "X", Nichrome, Nichrome V, and Ilium G in an aqueous solution containing 18 (± 1) ounces of sodium hydroxide (NaOH), 18 (± 1) ounces of sodium carbonate (Na_2CO_3), and 7 to 11 ounces of potassium permanganate (KMnO_4) per gallon of solution at a temperature of 180 to 190° F for 2 ($\pm 1/4$) hours. Upon removal, rinse thoroughly in tap water then immediately immerse in a second aqueous solution containing 6 (± 1) percent sulphuric acid (66° Baume) by volume and 1.5 (± 0.25) ounces of copper sulphate (CuSO_4) per gallon of solution at 180 to 190° F for a minimum of 1 hour. Rinse in room temperature tap water, neutralize in a 1 to 2 percent ammonia solution, then thoroughly rinse in demineralized water.

(d) Immerse Hastelloy A, Hastelloy B, and Hastelloy C in an aqueous solution containing 8.5 (± 0.5) percent sulphuric acid (66° Baume) by volume, 7.3 (± 0.3) ounces of sodium nitrate (NaNO_3), and 14.6 (± 0.6) ounces of sodium chloride (NaCl) per gallon of solution at 180 to 190° F for 30 to 90 minutes. Rinse thoroughly by immersion in tap water at 180° F ($\pm 10^\circ$), neutralize in a 1 to 2 percent ammonia solution, then thoroughly rinse in demineralized water.

3.5.4.7 Copper and copper-base alloys - Immerse in an aqueous solution containing 60 to 75 percent sulphuric acid (66° Baume) by volume, 20 to 35 percent nitric acid (38° Baume) by volume, and 1/8 fluid ounce of hydrochloric acid (20° Baume) per gallon of solution at a temperature of 70 to 80° F for 5 to 15 seconds. Thoroughly rinse by immersion in tap water at 70 to 80° F. Immerse in another aqueous solution containing 4 to 6 ounces of sodium cyanide (NaCN) and 2 to 4 ounces of sodium hydroxide (NaOH) per gallon of solution at 70 to 80° F for 15 to 30 seconds. Thoroughly rinse by immersion in demineralized water at 70 to 80° F.

3.5.5 Sensitization - After cleaning or pre-treating as described in paragraphs 3.5.3 and 3.5.4 above, metals and alloys shall be sensitized in the applicable manner specified below.

3.5.5.1 Magnesium and magnesium alloys - After cleaning and pre-treating as specified in paragraphs 3.5.3.2 and 3.5.4.1 above, magnesium and its alloys for all practical purposes have a nonmetallic, conversion-coated surface. Immerse such surfaces in an aqueous solution containing 3.5 to 4.5 percent hydrochloric acid (38° Baume) by volume and 40 (± 2) grams of stannous chloride (SnCl_2) per gallon of solution for 1 to 3 minutes at 70 to 80° F. Do not rinse the sensitized metal after removal from the SnCl_2 bath.

3.5.5.2 Zinc base or high zinc alloys

- Copper strike in accordance with Table 4-21.
- Pickle in the manner specified for copper in paragraph 3.5.4.7.
- Sensitize by immersion in an aqueous solution containing 10 (\pm 1) percent hydrofluoric acid (70 percent) by volume for 5 to 30 seconds at 70 to 80° F. Rinse thoroughly with demineralized water.

3.5.5.3 Aluminum and its alloys - No sensitization required after the pickling operation specified in paragraph 3.5.4.3.

3.5.5.4 Iron-base alloys

A. Iron and low-carbon steels - No sensitization required after the pickling operation specified in paragraph 3.5.4.4.A.

B. High carbon and tool steels - Sensitize in the manner outlined in paragraph 3.5.5.2 (c).

C. Stainless steels - Sensitize in the manner outlined in paragraph 3.5.5.2 (c).

3.5.5.5 Cobalt and cobalt-base alloys - No sensitization required after the pickling operation specified in paragraph 3.5.4.5.

3.5.5.6 Nickel and nickel-base alloys - No sensitization required after the pickling operation specified in paragraph 3.5.4.6.

3.5.5.7 Copper and copper base alloys - Sensitize copper and copper alloys in the manner outlined in paragraph 3.5.5.2 (c).

3.5.6 Activation - After cleaning, and pre-treating and sensitization as described in paragraphs 3.5.3, 3.5.4, and 3.5.5 above, metals and alloys shall have their surfaces activated as specified below.

3.5.6.1 Magnesium and magnesium alloys

(a) Immerse in an aqueous solution containing 0.25 to 0.30 percent hydrochloric acid (38° Baume) by volume and 1.0 to 1.5 grams of palladium chloride (PdCl_2) per gallon of solution at a temperature of 125 to 150° F and a pH of 4.0 to 5.0 for 1 to 3 minutes.

TABLE 4-21. . COPPER STRIKE BATHS

Bath Constituents and Operating Conditions	Aluminum Bath 1	Magnesium Bath 2
Copper cyanide, oz/gal	5.5	5.5
Sodium cyanide, oz/gal	6.5	6.8
Sodium carbonate, oz/gal	4.0	4.0
Rochelle salt, oz/gal	8.0	6.0
By analysis		
Copper, oz/gal	3.9	3.9
Free sodium cyanide, oz/gal	0.5	0.75
Conditions		
pH, Colorimetric	10.2-10.5	9.6-10.4
Temperature, °F	100-130	150-160

(b) Rinse thoroughly in demineralized water at 70 to 80° F upon removal from the activator bath.

3.5.6.2 Zinc base or high-zinc alloys - Activate in manner specified in paragraphs 3.5.6.1 (a) and (b).

3.5.6.3 Aluminum and its alloys - No activation required after the pickling operation specified in paragraph 3.5.4.3.

3.5.6.4 Iron-base alloys

A. Iron and low-carbon steels - No activation required after the pickling operation specified in paragraph 3.5.4.4.A.

B. High-carbon and tool steels - Activate in the manner outlined in paragraphs 3.5.6.1 (a) and (b).

C. Stainless steels - Activate in the manner outlined in paragraphs 3.5.6.1 (a) and (b).

3.5.6.5 Cobalt and cobalt-base alloys - No activation required after the pickling operation specified in 3.5.4.5.

3.5.6.6 Nickel and nickel-base alloys - No activation required after the pickling operation specified in 3.5.4.6.

3.5.6.7 Copper and copper base alloys - Activate copper and copper base alloys in the manner outlined in paragraphs 3.5.6.1 (a) and (b).

3.5.7 Masking - Components requiring only partial or special-area plating shall be masked with Minnesota Mining and Manufacturing Company No. 470 Electroplaters Tape, or equal, over those areas which do not require plating.

3.6 Plating Operations

3.6.1 General - With most basis materials, electroless nickel plating shall be deposited directly on the basis materials without a preliminary plating of any other metal. Surfaces made of or contaminated by the materials specified in paragraph 1.3 shall be given a copper flash by the electrolytic method then plated with electroless nickel as though they were pure copper (see Table 4-21).

3.6.2 Metals and alloys - Metallic components prepared for plating in conformance with paragraphs 3.5.3 through 3.5.7 shall be plated to the specified thickness (see paragraph 3.6.3) by immersion in an electroless nickel

plating bath of the specified composition (see Table 4-19) operated under the conditions specified in Table 4-19. Parts shall remain in the bath for a period of time sufficient to allow deposition of the required thickness of nickel coating, based on the deposition rate shown in Table 4-19 for the plating bath used (see Table 4-19).

NOTE: All of the plating baths specified in Table 4-19 are suitable for use with any of the basis metals covered by this specification. The selection of a bath for use therefore depends only upon the desired rate of plating and desired brightness of final deposit.

3.6.3 Thickness of plating - Plating thickness shall in all cases conform to the requirements of the applicable Engineering drawing or Engineering order. Unless otherwise specified, the thickness of nickel plating shall be as specified on all visible surfaces which can be touched by a ball 0.75 inch in diameter.

3.6.3.1 Minimum thickness when not specified on drawing - The minimum thickness of electroless nickel plate shall be 0.0005 inch except on:

- Externally threaded portions of articles where the tolerances of the thread preclude deposition of 0.0005 inch but, unless otherwise specified, not less than 0.00025 inch thick.

- Parts whose dimensional tolerances will not permit a thickness of 0.0005 inch but shall not be less than a maximum compatible with the dimensional tolerances.

- Washers, studs, bolts, nuts and other articles with major portions externally threaded but, unless otherwise specified, shall be not less than 0.00025 inch thick.

3.6.3.2 Maximum thickness when not specified on drawing - Where a dimensional plating tolerance is not specified, the maximum thickness shall be that which does not interfere with the function of the part; however, no plating over 0.005 inch thick shall be applied except on approval by the MSFC initiating activity.

3.7 Postplating Operations

3.7.1 Neutralizing - Plating solutions entrapped in parts having complicated shapes, channels, dead-end holes, etc., shall be neutralized by an alkaline solution containing from 2 to 5 percent sodium carbonate.

3.7.2 Water-spot prevention - Water spots shall be eliminated by immersion of plated parts in a 1 percent by volume potassium dichromate solution for 1 to 2 minutes.

3.7.3 Rinsing and drying - After plating all parts shall be promptly rinsed in clean, flowing water, then thoroughly dried.

3.7.4 Adhesion improvement - In order to improve the adhesion of electroless nickel plating to aluminum and aluminum alloys, after plating these metals shall be heated in an inert atmosphere, vacuum, or air furnace for 1 hour within the temperature range of 295 to 305° F.

3.7.5 Corrosion-resistance improvement - In order to improve the general corrosion resistance of electroless nickel plating on any metal specified in paragraph 1.2, plated parts, whose mechanical properties will not be adversely affected by the baking temperature involved, shall be heated in an inert atmosphere or vacuum furnace for 1 hour within the temperature range of 300 to 450° F.

3.7.6 Improvement of wear or abrasion properties - Metals and alloys, which would not be adversely affected by the baking temperatures involved, shall be heated in an inert atmosphere, vacuum furnace, or air furnace for 1 hour within the temperature range of 500 to 935° F. Hardness of the electroless nickel plating after this baking operation shall be a minimum of 700 Vickers.

3.7.7 Hydrogen embrittlement relief

3.7.7.1 Electroless nickel plated steel - Within 1 hour after the completion of the nickel plating operation, steel components shall be placed in a furnace and heated in air in the manner specified below:

A. Steel having a hardness of Rockwell C-35 to C-40 - Parts having a hardness of Rockwell C-35 to C-40 shall be heated to 375° F ($\pm 25^\circ$) and maintained at temperature for a minimum of 4 hours.

B. Steel having a hardness greater than Rockwell C-40 - Parts having hardnesses greater than Rockwell C-40 shall be heated to 375° F ($\pm 25^\circ$) and maintained at temperature for a minimum of 24 hours.

3.7.8 Plating removal (Stripping) - Remove electroless nickel plating by immersing the dry, plated parts in an aqueous solution containing 20 to 50 percent nitric acid (38° Baume) by volume and maintained at a maximum temperature of 95° F. Parts shall be left in the acid solution until all plating has dissolved.

3.8 Adhesion of Plating - Adhesion of electroless nickel plating shall be such that, when examined at a magnification of 4 diameters, it does not show separation from the basis metal at the interface after testing as specified in paragraph 4.3.2. The interface between the nickel and the basis metal is defined as the surface of the basis metal before plating. Cracks in the nickel plate, caused by bending and rupture of the basis metal, which do not result in flaking, peeling or blistering of the plate shall not be considered as lack of adhesion.

3.9 Corrosion Resistance - Electroless nickel plating meeting the minimum thickness requirements of paragraphs 1.4 and 3.6.3 and deposited on substrates requiring corrosion protection, shall show no corrosion products after 48 hours continuous exposure to synthetic sea-water salt spray.

3.10 Plating Hardness

3.10.1 As deposited - As-deposited electroless nickel coatings shall have a hardness within the range of 400 to 600 Vickers.

3.10.2 After heat treatment - Electroless nickel coatings baked in the manner specified in paragraph 3.7.6 shall have a minimum hardness of 700 Vickers.

4. QUALITY ASSURANCE PROVISIONS

4.1 Test Specimens - When plated articles are of such form as to be not readily adaptable to a test specified herein, necessary tests shall be made using separate specimens plated with the articles they represent. Prior to plating, separate specimens shall be of the same basis metal, of the same form (casting, wrought or forged), and of the same surface finish as the articles they represent, except that hot rolled specimens may be used to represent cast or forged articles. Separate specimens shall be approximately 1 inch wide, 4 inches long, and 0.040 inch thick for adhesion tests; but shall be at least 4 inches wide, 6 inches long and approximately 0.040 inch thick for all other tests. Separate specimens shall be introduced into a group of parts at regular intervals prior to the cleaning operations preliminary to plating and shall not be removed therefrom until after completion of the plating process. Conditions affecting the plating of the specimens, including the spacing and positioning with respect to other objects being plated, shall correspond as nearly as possible to those affecting the significant surfaces of the articles represented.

4.2 Sampling

4.2.1 Lot - For the purposes of this specification, a lot is defined as a group of plated articles of the same size, the same shape, having electroless nickel plating, and submitted for inspection at the same time.

4.2.2 Sampling for visual inspection and for non-destructive tests of plating thickness - A sample shall be drawn from each inspection lot by taking at random from the lot the percentage of articles specified in Table IV. These articles shall then be subjected to both the visual inspection and the plating thickness determination outlined in paragraphs 4.2.2.1 and 4.2.2.2, respectively.

4.2.2.1 Visual examination - All of the sample articles selected in paragraph 4.2.2 shall be visually examined to assure conformance to all the requirements of Section 3. If the number of nonconforming articles exceeds the acceptance number for that size sample (see Table 4-22), the lot represented by that sample shall be rejected.

4.2.2.2 Thickness determination - All of the sample articles selected in paragraph 4.2.2 shall have their plating thickness measured by a non-destructive method at several locations on each article where the plating is expected to be of minimum thickness. An article shall be considered to be nonconforming if any measurement fails to meet the specified minimum thickness. If the number of nonconforming articles in any sample exceeds the acceptance number for that size sample (see Table 4-22), the lot represented by that sample shall be rejected.

4.2.3 Sampling for destructive tests of plating thickness, adhesion, and salt spray resistance - A random sample, of the size specified in Table 4-23, but composed of different articles than those selected for non-destructive tests in paragraph 4.2.2, shall be selected from each inspection lot. An additional eight separate test specimens (see paragraph 4.1) shall be prepared by plating at the same time as the articles in the inspection lot to represent each inspection lot.

4.2.3.1 Thickness tests - Each article of the sample selected in paragraph 4.2.3 shall be tested in several locations at which the plating would be expected to be a minimum. If the plating thickness at any location on any article is less than the specified thickness, the lot represented by that article shall be rejected.

4.2.3.2 Salt spray resistance - For electroless nickel plating, four of the separate test specimens required in paragraph 4.2.3 shall be tested in lieu of plated articles which comprise the inspection lot. The failure of any test specimen in less than the time specified shall result in the rejection of the lot represented by that test specimen.

4.2.3.3 Adhesion - All the articles used for the plating thickness tests, if of suitable size and form, shall be used as specimens for the adhesion tests. If the articles used for plating thickness tests are not of suitable size and form, an equal number of additional articles shall be selected, or the four remaining

TABLE 4-22. SAMPLING FOR NON-DESTRUCTIVE TESTS

ITEMS IN INSPECTION LOT (NUMBER)	ITEMS IN SAMPLE (PERCENT OF INSPECTION LOT)		ACCEPTANCE NUMBER (MAXIMUM ALLOWABLE NUMBER OF ITEMS NONCONFORMING)
	Visual (4. 2. 2. 1)	Thickness (4. 2. 2. 2)	
1 to 6	100	100	0
7 to 15	100	100	0
16 to 40	100	100	0
41 to 110	100	60	0
111 to 300	60	40	1
301 to 500	30	20	1
501 and over	20	15	2

TABLE 4-23. SAMPLING FOR DESTRUCTIVE TESTS

ITEMS IN INSPECTION LOT (NUMBER)	ITEMS IN SAMPLE (NUMBER)	ACCEPTANCE NUMBER (MAXIMUM ALLOWABLE NUMBER OF ITEMS NONCONFORMING)
1	0	0
2 to 5	1	0
6 to 25	3	0
26 to 100	4	0
101 to 300	6	0
301 to 5000	10	0
5001 and over	15	0

separate test specimens (see paragraphs 4.2.3 and 4.2.3.2) may be used. Failure of any test specimen shall result in the rejection of the lot represented by that test specimen.

4.2.3.4 Hydrogen embrittlement relief - A random sample of the size specified in Table V shall be selected from each lot of plated steel articles and tested for hydrogen embrittlement relief in the manner specified in paragraph 4.3.4. Failure of any of the articles tested shall result in the rejection of the lot represented by that article.

4.2.3.5 Hardness - Each of the articles selected in paragraph 4.2.3 shall be tested for hardness in several different locations on the specimen. If the plating hardness at any location is less than the specified hardness, the lot represented by that article shall be rejected.

4.3 Test Methods

4.3.1 Thickness - Thickness determinations shall be made by the microscopic method, or by an instrument or electrochemical method approved by the MSFC initiating activity. For referee tests, the microscopic method shall be used. Measurement of minimum plating thickness on bolts shall be made on the shank adjacent to the thread, or on the head if the shank is completely threaded.

4.3.1.1 Microscopic test

A. Specimens - Specimens for microscopic measurements shall consist of cross-sections of plated articles, or test specimens, suitably mounted, ground, polished, and etched to differentiate the nickel plating from the basis metal.

B. Metallurgical microscope - The plating thickness of prepared specimens may be measured by means of a metallurgical microscope equipped with a filar micrometer ocular which has been calibrated against a standard scale to an accuracy of plus or minus 2 percent.

C. Metallographic microscope - The plating thickness of prepared specimens may be measured by means of a metallographic microscope by projecting the image of the specimen on the ground-glass focusing plane of the camera at a known magnification. The actual plating thickness can then be easily determined by measuring the plating thickness of the projected image in inches, and dividing this measured value by the known image magnification.

4.3.2 Adhesion

4.3.2.1 Scrape method - Adhesion may be determined by scraping the surface of the plated article, or test specimen, to expose the basis metal. The junction of the plating and the exposed basis metal shall then be examined at four diameters magnification. Any evidence of flaking, blistering, or separating between the plating and the basis metal shall be interpreted as unsatisfactory adhesion.

4.3.2.2 Bend method - Adhesion may be determined by clamping the plated article, or test specimen, in a suitable holding device and rapidly bending at room temperature through an angle of 180 degrees, around a diameter equal to the thickness of the specimen, until plating rupture occurs. If the edge of the ruptured plating can be peeled back, or if separation between the plating and the basis metal can be seen at the point of rupture when examined at four diameters magnification, adhesion shall be considered as unsatisfactory.

4.3.3 Salt spray resistance - Salt-spray (synthetic sea water) resistance tests shall be made in the manner specified in Method 812 of Federal Test Method Standard Number 151a.

4.3.4 Hydrogen embrittlement relief (Steel parts only) - Hydrogen embrittlement relief tests shall be made by subjecting each of the plated steel articles in the lot sample (see paragraph 4.2.3) to the maximum tensile stress to which it is intended that the articles be exposed in actual service. This stress, as specified on the Engineering drawing or as directed by the MSFC procuring activity, shall be constantly maintained for a minimum period of 240 hours. The failure of any test specimen in less than 240 hours shall be interpreted as unsatisfactory hydrogen embrittlement relief of the lot represented by that specimen.

4.3.5 Hardness Hardness measurements shall be made, using steel test specimens having a minimum of 3 mils plating thickness, on a Vickers machine with a diamond pyramidal indenter and employing a 1-kilogram load.

4.4 Retests - Plated articles which have been subjected to a chemical method of plating thickness determination, and articles rejected or withdrawn due to defective or deficient plating, may in both cases be resubmitted after stripping (see paragraph 3.7.8) and replating of the individual articles in the first case, and of the entire lot in the second case.

4.5 Plating Solution Analyses

4.5.1 Copper in copper strike bath - Analyze in the manner outlined in paragraph 4.5.1 of Specification MS 120.4.

4.5.2 Cyanide in copper strike baths - Analyze in the manner outlined in paragraph 4.5.2 of Specification MS 120.4.

4.5.3 Nickel in electroless plating baths

- (a) Pipette a 5-ml sample into a 250-ml Erlenmeyer flask.
- (b) Using a graduated cylinder, add 70-ml of an ammonium chloride-sodium pyrophosphate-potassium iodide solution which is prepared by dissolving 54 g of ammonium chloride (NH_4Cl), 57 g of sodium pyrophosphate ($\text{Na}_4\text{P}_2\text{O}_7$), and 1.6 g of potassium iodide (KI) in warm water and diluting to 1 liter.
- (c) Add concentrated ammonium hydroxide drop by drop (approximately 3 drops) until the solution just faintly smells of ammonia. If too much ammonia is added, neutralize the excess with concentrated hydrochloric acid.
- (d) Fill a 50-ml burette with standard sodium cyanide solution and titrate until the turbidity, which forms at the beginning of the titration, disappears with the addition of a final drop of standard solution. The solution in the flask should then be perfectly clear.
- (e) Read the burette. This value multiplied by the factor for the standard sodium cyanide solution gives the ounces of metallic nickel per gallon of the plating bath.

4.5.3.1 Preparation of standard sodium cyanide solution

- (a) Dissolve 25 g of sodium cyanide (NaCN) and 1.7 g of silver nitrate (AgNO_3) in water and dilute to 1 liter.
- (b) Weigh about 0.2 g of pure nickel on an analytical balance and dissolve it in a mixture of 5 ml of sulphuric acid, 5 ml of nitric acid and 5 ml of water in a 250-ml Erlenmeyer flask.
- (c) Evaporate until dense white fumes of sulphur trioxide are evolved.
- (d) Cool, dilute with 10 ml of water, add a slight excess of ammonia and boil.
- (e) When the solution smells faintly of ammonia, cool to room temperature, add 75 ml of the chloride-phosphate solution and titrate with the sodium cyanide solution until the precipitate just redissolves.

(f) Record the ml of standard sodium cyanide solution used.

(g) Calculate the factor as follows:

$$\frac{\text{Wt of pure Ni X 200}}{\text{ml of standard NaCN X 7.5}} = \text{Factor in oz/gal for metallic nickel}$$

4. 5. 4 Hypophosphite in electroless plating baths

(a) Dilute a 25 ml sample of the acid electroless plating solution to 1 liter.

(b) Take a 5 ml aliquot.

(c) Add 10 ml of 10 percent ammonium molybdate solution.

(d) Add 10 milliliters of 6 percent sulphurous acid.

(e) Cover with watch glass and heat to boiling. A deep blue color should develop.

(f) Cool to room temperature.

(g) Dilute to 100 ml.

(h) Determine transmittancy at 440 m .

The calibration curve is linear when plotted on semilog paper.

4. 5. 4. 1 Preparation of ammonium molybdate solution - Dissolve 65 grams of ammonium heptamolybdate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$), 25 grams of ammonium nitrate (NH_4NO_3), and 15 ml of concentrated ammonium hydroxide in 600 ml of water. Filter and dilute to 1 liter.

4. 5. 5 Phosphate in electroless plating baths

(a) Add 20 ml of 5 percent sodium bicarbonate solution to 10 ml of the acid plating solution to make the solution alkaline. .

(b) Cool in ice bath.

(c) Add 50 ml of 0.1 N standardized iodine solution.

- (d) Stopper flask securely.
- (e) Let stand at room temperature for at least 2 hours.
- (f) Cool in ice bath for 15 minutes.
- (g) Acidify with acetic acid.
- (h) Titrate excess iodine with 0.1 N standardized sodium thiosulphate solution using starch indicator near end point.
- (i) Calculate as follows:

$$\text{Na}_2\text{HPO}_3 \text{ in grams per liter equals } \frac{0.1 \text{ N iodine, net ml}}{\text{ml of plating solution}} \times 6.3$$

4. 5. 5. 1 Preparation of 0.1 N standard iodine solution

- (a) Dissolve 24 g of analytical-grade potassium iodide (KI) in 175 ml of water.
- (b) Add 12.8 g of analytical-grade iodine, stir the solution until the iodine has dissolved then dilute to 1 liter with distilled water.
- (c) Store the iodine solution in a stoppered brown-glass bottle away from strong sunlight.
- (d) On an analytical balance accurately weigh about 0.15 g of pure dry arsenious oxide (As_2O_3), dissolve it in 20 ml of 1 N sodium hydroxide solution, and add 15 ml of 2 N sulphuric acid.
- (e) Carefully add 4 g of sodium bicarbonate (NaHCO_3) dissolved in 75 ml of distilled water.
- (f) Add 2 ml of starch solution to the arsenious oxide solution.
- (g) Fill a 50-ml burette with standard iodine solution and titrate the arsenious oxide solution to a blue end point.
- (h) Read the burette.

(i) Calculate as follows:

$$\frac{\text{Wt of As}_2\text{O}_3 \times 1.2 \times 200}{\text{ml of standard I}_2 \times 7.5} = \text{Factor in oz/gal for metallic Sn}$$

4.5.5.2 Preparation of 0.1N standard sodium thiosulphate solution

(a) Prepare and standardize sodium thiosulphate solutions in the manner outlined in paragraph 4.7.1 (j) of specification MS 120.5.

(b) Using distilled water, dilute solutions obtained in (a) to the necessary volume to result in a 0.1N solution.

5. PREPARATION FOR DELIVERY

Not applicable to this specification.

6. NOTES

6.1 Intended Use - This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use in electroless nickel plating of materials for use in space launch vehicle applications.

6.2 Important Considerations

6.2.1 Temperature control - Electroless nickel plating solutions decompose when in contact with surfaces hotter than 284° F so the heating method used must not allow the solution to reach this temperature even at heat exchanger surfaces.

6.2.2 pH adjustment - The most important plating bath parameter is pH which should always be adjusted by adding pure dilute solutions of reagent grade chemicals while stirring or mechanically agitating the bath and at temperatures lower than 140° F.

6.2.3 Makeup water - Only distilled or deionized water should be used for makeup purposes in electroless nickel processing baths.

6.2.4 Filtration - Even when reagent grade chemicals are used, new electroless nickel plating baths should be filtered at least once through activated carbon then dummied electrolytically (about 10 amp-hr/gal) prior to use.

NOTICE: When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING SPECIFICATION FOR
COPPER PLATING (ELECTRODEPOSITED)

1. SCOPE

1.1 Scope — This manufacturing specification covers the approved PE Laboratory methods and requirements for the copper plating of components to be used in space launch vehicle applications.

1.2 Applicability — The methods and requirements set forth in this specification are applicable to copper plating of carbon steels, stainless steel, aluminum, copper, magnesium, titanium, and their alloys.

1.3 Classification — Copper plating shall be of the following classes as specified.

Class 1 - 0.00100 inch thick minimum.

Class 2 - 0.00050 inch thick minimum.

Class 3 - 0.00020 inch thick minimum.

Class 4 - 0.00010 inch thick minimum.

2. APPLICABLE DOCUMENTS

2.1 Government - The following documents of the issue in effect on the date of use of this specification, form a part of this specification.

SPECIFICATIONS

Federal

0-A-451d (1)	Ammonia
O-C-823	Copper Sulfate
O-P-557	Potassium Hydroxide
O-S-377	Tetrasodium Pyrophosphate

O-S-581d	Sodium Carbonate
O-S-598	Sodium Hydroxide (technical)
O-S-809a	Sulfuric Acid (technical)
<u>Military</u>	
MIL-C-14550	Copper Plating (Electrodeposited)
MIL-C-22045	Copper Anodes

George C. Marshall Space Flight Center

MS100.2A	Manufacturing Specification for the Deoxidation of Aluminum Alloys
MS100.4A	Manufacturing Specification for the Cleaning and Passivation of Austenitic (300 Series) Stainless Steel Suction Lines
MS101.3	Manufacturing Specification for the Cleaning of Carbon Steel Alloys
MS120.0	Manufacturing Specification for Cadmium Plating of Aluminum, Copper, and Steel Alloys
MS150.0	Manufacturing Specification for Vapor Degreasing of Metallic Surfaces
MS150.4	Manufacturing Specification for Electro-polishing of Corrosion Resistant Steel

2.2 Other Publications — The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

Society of Automotive Engineers, Inc.

A. M. S. 2418A Copper Plating

(Copies of the publication listed above may be obtained from the Society of Automotive Engineers, Inc., 485 Lexington Ave., New York 17, N. Y.)

Electroplating Engineering Handbook 2nd Edition

(Copies of this publication may be obtained from the Reinhold Publishing Corporation, 430 Park Ave., New York 22, N. Y.)

Chemicals such as the following may be procured from other sources or equivalent.

Fisher Chemical Index 62-C

C-460	Copper Cyanide
S-283	Sodium Cyanide (Technical)
S-384	Rochelle Salt
Z-52	Zinc Oxide
Z-68	Zinc Sulphate
P-237	Potassium Fluoride
	Copper Pyrophosphate
	Copper Fluoborate
	Fluoboric Acid

(Copies of this publication may be obtained from the Fisher Scientific Company, 711 Forbes Ave., Pittsburgh, Pa.)

Metal Finishing Guidebook

(Copies of this publication may be obtained from the Metals and Plastics Publications, 381 Broadway, Westwood, New Jersey.)

3. REQUIREMENTS

3.1 General — No deviation from the requirements of this specification shall be permitted without prior written approval of the MSFC initiating activity.

3.2 Fabrication Operations Prior to Plating — Unless otherwise specified by the Engineering drawing, the plating shall be applied after all basis metal heat treatments and mechanical operations on the part such as machining, brazing, welding, forming, and perforating have been completed.

3.3 Appearance — Copper plating shall be smooth, uniformly deposited, fine grained, adherent, with no visible blisters, pits, and indications of burning; and with no other detrimental defects. Slight staining or discoloration will not be cause for rejection.

3.4 Equipment

3.4.1 Plating tanks — Tanks of a suitable size, constructed of heavy gauge hot rolled steel sheets with joints welded inside and out shall be used for containing basic cleaning and plating solutions. Sulfuric acid solutions shall be contained in lead lined steel tanks.

3.4.2 Bars, hangars, racks, and baskets — Tanks shall be equipped with proper types of bars, hangars, racks and baskets to assure an efficient cleaning and plating operation.

3.4.2.1 Aluminum alloys — Aluminum racks shall be used in the plating of aluminum alloys. Aluminum racks shall have spines fabricated from 1100 alloy and contacts fabricated from 2024 alloy.

3.4.2.2 Copper and steel alloys — Copper or steel racks shall be used in the plating of copper and steel alloys respectively.

3.4.2.3 Magnesium — Conventional non-magnesium racks may be used. Organic rack coatings shall be used to confine the exposed rack area to the minimum necessary for electrical contact.

3.4.2.4 Titanium — Titanium racks are recommended for the anodic etch process, described in paragraph 3.6.3.

3.5 Materials

3.5.1 General — The materials such as chemicals, anodes, and cleaning agents shall produce coatings which meet the requirements of this specification and which will have no deleterious effect on the corrosion resistance of the basis metal.

3.5.2 Chemicals — The chemicals used in the preparation of copper plating solutions shall be chemical pure (C.P.) grade or best quality technical grade.

3.5.3 Water — The water used in the preparation of copper plating solutions shall be demineralized water having a minimum specific resistance of 50 000 ohms.

3.5.4 Basic metal — The basic metal shall be free from visible defects that will be detrimental to the appearance or the performance of the plating.

3.6 General Procedure

3.6.1 Pre-plating requirements — All uncarburized steel parts having a hardness greater than Rockwell C 40 shall be stress relieved prior to cleaning or plating by heating at 375° F ($\pm 25^\circ$) for 3 hours. Stress relief baking should not reduce the hardness of the steel parts to less than the specified minimum.

3.6.1.1 General — All surfaces to be plated shall be thoroughly cleaned prior to the application of the copper plate. Meticulous cleaning and surface preparation of the components cannot be overemphasized since this factor is of prime importance in obtaining a satisfactory adherence.

3.6.1.2 Aluminum — Aluminum and its alloys shall be cleaned in the manner outlined in specification MS 100.2A.

3.6.1.3 Carbon steels — Carbon steels shall be cleaned and prepared for plating in the manner outlined in paragraphs 3.4.4.2 and 3.4.4.3 of Specification MS 120.0.

3.6.1.4 Stainless steels — Stainless steels shall be cleaned and prepared for plating in the manner outlined in Specifications MS 100.4A and MS 150.4.

3.6.1.5 Titanium — Titanium and its alloys shall be cleaned in the manner outlined in Specification MS 150.0.

3.6.1.6 Copper and copper alloys — Copper and copper alloys shall be cleaned and prepared for plating in the manner outlined in paragraph 3.4.4.5 of Specification MS 120.0.

3.6.1.7 Magnesium — Magnesium and its alloys shall be cleaned and prepared for plating as outlined in Table 4-24.

3.6.2 Zinc immersion

3.6.2.1 Aluminum and aluminum alloys — Aluminum and its alloys shall be given a zinc-immersion treatment according to Table 4-25 prior to copper plating.

3.6.2.2 Magnesium — Magnesium shall be given a zinc-immersion treatment according to Table 4-26 prior to copper plating.

3.6.3 Anodic etch — Titanium shall be given an anodic etch treatment according to Table 4-27 prior to copper plating.

3.6.4 Plating

3.6.4.1 General — After proper cleaning, carbon steel, stainless steel, and copper alloys shall be copper plated directly without a preliminary plating of any other metal. Aluminum and its alloys, and magnesium require a zinc-immersion deposit prior to copper plating. Titanium requires an anodic etch treatment prior to copper plating.

3.6.4.2 Aluminum and aluminum alloys — Aluminum and its alloys shall be given a copper strike according to bath 1 of Table 4-28 followed by plating in one of the baths as set forth in Table 4-29 or 4-30.

3.6.4.3 Magnesium and magnesium alloys — Magnesium and its alloys shall be given a copper strike according to bath 2 of Table 4-28, followed by plating in one of the baths as set forth in Table 4-29 or 4-30.

3.6.4.4 Stainless steel and carbon steel — Stainless steel and carbon steel components shall be copper plated (electro-deposited) in a plating bath of the specified composition, at the specific temperature, and allowing it to remain in the bath for a period of time sufficiently long to allow the deposition of the copper coating. These components can be copper plated by any method as set forth in Tables 4-29 or 4-30.

3.6.4.5 Titanium — Titanium shall be copper plated by the method as set forth in Table 4-31.

3.7 Thickness of Plating — Unless otherwise specified the thickness of copper plate shall be as indicated in paragraph 1.3 on all visible surfaces which can be touched by a ball 0.75 inch in diameter.

3.8 Hydrogen Embrittlement Relief

3.8.1 Copper plated steel — Within one hour after the completion of the copper plating operation, steel components shall be placed in a furnace and heated in air in the manner specified below. Plated articles need not be heat treated if the pretreatment and plating processes used have been demonstrated not to have harmfully affected their performance.

3.8.1.1 Parts having a hardness of Rockwell C-35 to C-40 — Parts having a hardness of Rockwell C-35 to C-40 shall be heated to 375° F (±25°) and maintained at temperature for a minimum of 4 hours.

3.8.1.2 Parts having a hardnesses greater than Rockwell C-40 — Parts having hardnesses greater than Rockwell C-40 shall be heated to 375° (±25°) and maintained at temperature for a minimum of 24 hours.

4. QUALITY ASSURANCE PROVISIONS

4.1 General — To determine adequate control over the quality of plating, weekly analyses shall be made of the composition of the plating bath and other processing baths. Verification of analyses and tests, in the form of recorded data, shall be maintained. The results of all tests for adhesion, thickness, corrosion, and plating bath analyses shall be recorded.

4.2 Sampling

4.2.1 Lot — A lot shall consist of plated articles of the same class, type, and condition, and of approximately the same size and shape and plated under similar conditions, submitted for inspection at one time.

4.2.2 Sampling for visual inspection and for non-destructive tests of plating — A sample shall be drawn from each inspection lot by taking at random, from the lot, the percentage of articles specified in Table 4-32.

4.2.2.1 Visual examination — All sample articles selected shall be visually examined to assure conformance to the requirements of paragraph 3.3. If the number of nonconforming articles exceeds the acceptance number for that size sample (see Table 4-32, the lot represented by that sample shall be rejected).

4.2.2.2 Thickness determination — All of the sample articles selected shall have their plating thickness measured by a non-destructive method at several locations on each article where the plating is expected to be of minimum thickness. An article shall be considered to be non-conforming if any measurements fails to meet the specified minimum thickness. If the number of non-conforming articles in any sample exceeds the acceptance number for that size sample (Table 4-32), the lot represented by that sample shall be rejected.

4.2.3 Sampling for destructive tests of plating — A random sample of the size specified in Table 4-33, but composed of different articles than those selected for nondestructive test in paragraph 4.2.2 shall be selected from each inspection lot and eight separately plated test specimens shall be prepared to represent each inspection lot.

4.2.3.1 Thickness tests — The thickness of the copper shall be as specified on all visible surfaces which can be touched by a ball 0.75 inch in diameter. If the plating thickness is less than specified, the lot represented by that article shall be rejected.

4.2.3.2 Adhesion — All of the articles used for the plating thickness tests, if of suitable size and form, shall be used as specimens for the adhesion tests. If the articles used for plating thickness tests are not of suitable size and form, an equal number of additional articles shall be selected or separately plated test specimens used. Failure of any test specimen shall result in the rejection of the lot represented by that test specimen.

4.2.3.3 Hydrogen embrittlement relief — Each steel article of the sample selected in paragraph 4.2.3 shall be tested for hydrogen embrittlement relief. The failure of any test article shall be interpreted as unsatisfactory hydrogen embrittlement relief of the lot represented by that article.

4.3 Test Methods

4.3.1 Thickness - Thickness determinations shall be made by the microscopic method, or by an instrument or electrochemical method approved by the MSFC initiating activity. For referee tests, the microscopic method shall be used.

4.3.1.1 Microscopic test

A. Specimens - Specimens for microscopic measurements shall consist of cross-sections of plated articles, or test specimens, suitably mounted, ground, polished, and etched to differentiate the copper plating from the basis metal.

B. Metallurgical microscope - The plating thickness of prepared specimens may be measured by means of a metallurgical microscope equipped with a filar micrometer ocular which has been calibrated against a standard scale to an accuracy of ± 2 percent.

C. Metallographic microscope - The plating thickness of prepared specimens may be measured by means of a metallographic microscope by projecting the image of the specimen on the ground glass focusing plane of the camera at a known magnification. The actual plating thickness can then be easily determined by measuring the plating thickness of the projected image in inches, and dividing this measured value by the known magnification.

4.3.2 Adhesion

4.3.2.1 Scrape method - Adhesion may be determined by scraping the surface of the plated article, or test specimen, to expose the basis metal. The junction of the plating and the exposed basis metal shall then be examined at four diameters magnification. Any evidence of flaking, blistering, or separation between the plating and the basis metal shall be interpreted as unsatisfactory adhesion.

4.3.2.2 Bend method - Adhesion may be determined by clamping the plated article, or test specimen, in a suitable holding device and rapidly bending at room temperature through an angle of 180 degrees, around a diameter equal to the thickness of the specimen, until plating rupture occurs. If the edge of the ruptured plating can be peeled back or if separation between the plating and the basis metal can be seen at the point of rupture when examined at four diameters magnification, adhesion shall be interpreted as unsatisfactory.

4.3.3 Hydrogen embrittlement relief — Hydrogen embrittlement relief tests shall be made by subjecting each of the plated susceptible steel articles in the lot sample to the maximum stress to which it is intended that the articles be exposed in actual service. This stress shall be specified by the MSFC Procuring Activity and shall be constantly maintained for a minimum period of 240 hours. The failure of any test article in less than 240 hours shall be interpreted as unsatisfactory hydrogen embrittlement relief of the lot represented by that article.

4.4 Retests — Plated articles which have been subjected to a chemical method of plating thickness determination and articles rejected or withdrawn due to defective or deficient plating may be resubmitted after stripping and replating of the individual articles in the first case and of the entire lot in the second case.

4.5 Analysis of Copper and Free Cyanide in Copper Strike Bath

4.5.1 Copper - Proceed as follows:

(a) Fill the sampling burette with the solution to be tested, and run a 20-ml sample into a 250-ml Erlenmeyer flask.

(b) Add 5 ml of concentrated sulfuric acid and 2 to 3 ml of concentrated nitric acid and boil until brown fumes are no longer expelled and the solution is clear. If considerable iron is present, the solution is difficult to clear. In this case add more nitric acid and heat the solution until all cloudiness disappears.

CAUTION: Perform operation under hood because poisonous fumes are given off.

(c) After cooling add 100 ml distilled water and then add concentrated ammonium hydroxide until a dark-blue color persists.

(d) Boil until most of the ammonia fumes are removed. Fifteen minutes of boiling is generally sufficient.

(e) Add 10 ml of 30-percent acetic acid.

(f) Cool to room temperature and replace any water lost in boiling.

(g) Add 15 ml of 30-percent potassium iodide and shake.

(h) Fill the 50-ml burette with standard sodium thio-sulfate and titrate until the dark brown color begins to fade.

(i) Add 5 ml of starch indicator and continue the titration until the blue color disappears for about one minute.

(j) Read the burette. This value multiplied by the factor for standard sodium thiosulfate gives the ounces of copper per gallon of the plating bath.

4.5.1.1 Preparation of Standard Sodium Thiosulfate Solution

Dissolve approximately 17.5 grams of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) and one gram of sodium hydroxide in 1 liter of distilled water.

The copper solution can be prepared by dissolving 40 grams of copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in one liter of water and acidifying with 10 ml of concentrated sulfuric acid. The copper is determined by electroanalysis of a 10-ml aliquot on a platinum cathode. A few drops of concentrated nitric acid added to the electrolyte will improve deposits.

An alternate method is to weigh about 2.5 grams of pure copper wire on analytical balance, dissolve the wire in 1:1 nitric acid, add 50 ml of concentrated sulfuric acid, and evaporate to sulfur trioxide fumes. Dilute to 100 ml in a volumetric flask. Standardize the sodium thiosulfate as follows:

(a) Pipette 10 ml of standard copper solution into a 250-ml Erlenmeyer flask.

(b) Add 100 ml of distilled water and concentrated ammonium hydroxide until a dark blue color persists.

(c) Boil until most of the ammonia fumes are removed (15 minutes).

(d) Add 10 ml of 30-percent acetic acid.

(e) Cool to room temperature and replace water lost in boiling.

- (f) Add 15 ml of 30-percent potassium iodide and shake.
- (g) Fill a 50 ml burette with the prepared sodium thiosulfate solution and titrate contents of flask until the dark brown color begins to fade.
- (h) Add 5 ml of starch indicator and continue titration until blue color disappears for one minute.

Calculate the factors as follows:

$$\frac{\text{Weight of Cu in 10 ml aliquot} \times 100}{\text{ml of standard Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O} \times 7.5} = \text{Factor in oz/gal for Metallic copper (Cu)}$$

4.5.1.2 Preparation of starch solution

Make a paste of 5 grams of soluble starch and 10 grams of zinc chloride, then add 150 ml of boiling water. Stir until dissolved and dilute to one liter, transfer to storage bottle.

4.5.2 Free sodium or potassium cyanide - Proceed as follows:

- (a) Fill the sampling burette with the solution to be tested and run a 10 ml sample into a 250 ml Erlenmeyer flask.
- (b) Add 100 ml of distilled water and with a dropper add one ml of a 10 percent potassium iodide indicator.
- (c) Fill the 50 ml burette with standard silver nitrate solution and titrate until a faint turbidity remains while shaking.
- (d) Read the burette. This value multiplied by the factor for standard silver nitrate solution gives either the oz/gal of free sodium or potassium cyanide in the plating bath.

4.5.2.1 Preparation of Standard Silver Nitrate Solution

Dissolve approximately 17 grams of silver nitrate (AgNO_3) in sufficient chloride-free distilled water to make 1 liter and store the solution in amber bottles.

The silver nitrate solution may be standardized against potassium chloride. Accurately weigh 0.3 gram of reagent grade potassium chloride which has been previously dried for 2 hours at 221°F. Carefully transfer the potassium chloride to a 250 ml Erlenmeyer flask and add 100 ml of distilled water and 2 drops of a 2 percent sodium chromate indicator solution. Fill a 50-ml burette with the silver nitrate solution and titrate until the first permanent reddish tint appears.

Calculations:

$$\frac{\text{Weight of KCL} \times 1.32 \times 100}{\text{ml of standard A} \times \frac{\text{NO}_3}{\text{g}} \times 7.5} = \text{Factor in oz/gal for free sodium cyanide (N}_a\text{CN)}$$

$$\frac{\text{Weight of KCL} \times 1.75 \times 100}{\text{ml of standard A} \times \frac{\text{NO}_3}{\text{g}} \times 7.5} = \text{Factor in oz/gal for free potassium cyanide (KCN)}$$

4.6 Acid Copper Sulfate Solutions

4.6.1 Sulfuric Acid - Proceed as follows

- (a) Pipette a 10 ml sample into a 250 ml Erlenmeyer flask.
- (b) Add 150 ml of distilled water and with a dropper add 10 drops of 0.1 percent Methyl Orange.
- (c) Fill the 50 ml burette with standard solution of 1.0 Normal sodium hydroxide and titrate until the color of the solution becomes a pale green.
- (d) Read the burette. This value multiplied by the factor for standard sodium hydroxide solution gives the oz/gal of sulfuric acid of the plating bath.

NOTE: Certain addition agents affect the color of this end point. If the color change cannot be seen easily, the solution may be further diluted and more indicator added or an activated carbon treatment of the sample may be employed.

4.6.1.1 Preparation of Standard (approximately 1.0 Normal Sodium Hydroxide Solution)

Weigh out 40 grams of reagent-grade sodium hydroxide (NaOH) and dissolve in about 500 ml of distilled water. Transfer the sodium hydroxide solution to a 1-liter volumetric flask and adjust to the mark with distilled water. The solution may be standardized as follows:

Accurately weigh out on an analytical balance 4 grams of potassium acid phthalate ($\text{KHC}_8\text{H}_4\text{O}_4$) which has been previously dried for 2 hours at 248°F. Transfer the potassium acid phthalate to a 250 ml Erlenmeyer flask and dissolve the salts in 100 ml of distilled water. Add 3 drops of a 1 percent alcoholic solution of phenolphthalein. Fill the 50 ml burette with the sodium hydroxide solution and titrate to a faint-pink color.

Calculate the factor as follows:

$$\frac{\text{Weight of KHC}_8\text{H}_4\text{O}_4 \times 0.24 \times 100}{\text{ml of standard NaOH} \times 7.5} = \text{Factor in oz/gal for sulfuric acid (H}_2\text{SO}_4 \text{ 100 percent).}$$

4.6.2 Total Copper Sulfate and Sulfuric Acid

- (a) Cool or warm the copper solution to 77°F.
- (b) Using a hydrometer graduated in 0.002 specific gravity units (0.2 degree Baume units) determine the specific gravity (Baume) of the solution.
- (c) Record the specific gravity of the solution to the nearest 0.001 division (0.1 degree Baume).
- (d) Using Table X and interpolating, if necessary, find the total oz/gal of copper sulfate plus sulfuric acid.
- (e) Subtracting the previously found oz/gal of sulfuric acid from the total oz/gal sulfuric acid plus copper sulfate gives the oz/gal of copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in the plating bath.

NOTE: The results obtained by this method will not be accurate if appreciable amounts of aluminum sulfate or other salts have been added to the bath. Quantities of iron or other impurities in the solution will also give high readings. If greater accuracy is required run a 10-ml sample into

a 250-ml Erlenmeyer flask and complete the analysis by starting with step (c) for copper under brass or bronze alloy solutions.

4.7 Copper Pyrophosphate Solutions

4.7.1 Copper

(a) Proceed as follows: Fill a sampling burette with the solution to be tested and run a 5-ml sample into a 250-ml Erlenmeyer flask.

(b) Add 10 ml of a 50 percent sulfuric acid solution and cautiously take a SO₂ fumes on a hotplate.

CAUTION: This operation should be performed under a hood since the SO₂ fumes are quite corrosive.

(c) Cool and dilute to 100 ml with distilled water. Heat 3 to 5 minutes and cool.

(d) Add 15 ml of a 30 percent potassium iodide solution.

(e) Fill a 50-ml burette with standard sodium thiosulfate and titrate until the color of the solution changes from a brown to a light yellow.

(f) Add 3 ml of starch indicator solution. (The starch indicator solution is prepared by thoroughly mixing 4 grams of soluble starch with 50 to 100 ml of cold water, dilute to approximately a liter with boiling water, if necessary, and stir until dispersed).

(g) Continue the titration until the blue color just disappears.

(h) Read the burette. This value multiplied by the factor for the standard sodium thiosulfate Na₂S₂O₃ gives the ounces of metallic copper per gallon of the plating bath.

Calculate the factor as follows:

$$\frac{\text{Weight of copper in 10 ml aliquot} \times 200}{\text{ml of Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O} \times 7.5} = \text{Factor in oz/gal for metallic copper.}$$

4.7.2 Pyrophosphate - Proceed as follows:

- (a) Pipette a 10-ml sample of the solution into a 250 ml volumetric flask.
- (b) Add about 100 ml of hot water (at least 122°F) and 10 ml of 400 g/l NaOH solution and shake.
- (c) Add 100 ml of the guanyurea sulfate reagent, shake the contents of the flask, and cool to room temperature.
- (d) Dilute the contents of the flask to the mark with water and shake.
- (e) Filter the supernatant liquid through a dry 15-cm No. 597 Sand-S paper (or equivalent), using a 10-cm ribbed funnel, into a dry 400-ml beaker. Collect about 225 ml of filtrate (6 to 10 minutes filtering time).
- (f) Pipette 100 ml of the filtrate into a 500-ml Erlenmeyer flask, add 3 to 4 drops of 1.0 percent phenolphthalein and concentrated hydrochloric acid, a drop at a time, until the red color changes to a light pink or becomes colorless.
- (g) Add 1 ml of bromcresol green indicator solution and immediately titrate with 0.5 Normal sulfuric acid until the color changes from blue to blue-green to yellow-green and finally to yellow.
- (h) Add 50 ml of a 125 g/l solution of zinc sulfate and allow to stand for 4 to 5 minutes with occasional swirling.
- (i) Allow standard 0.2 Normal sodium hydroxide solution to run in from a burette until the color changes from a light yellow to a blue-green and record the volume of sodium hydroxide used.
- (j) Calculate the oz/gal of pyrophosphate (P_2O_7) present according to the following equation:

$$\text{ml of standard NaOH} \times \text{factor F} = \text{oz/gal pyrophosphate (P}_2\text{O}_7\text{)}$$

$$\text{oz/gal P}_2\text{O}_7 \times 1.9 = \text{oz/gal potassium pyrophosphate (K}_4\text{P}_2\text{O}_7\text{)}$$

4.7.2.1 Preparation of Reagents and Standard Solutions for Pyrophosphate Analysis

A. Guanylurea Sulfate Reagent. Dissolve 40 grams of commercial guanylurea sulfate in about 750 ml of hot water. Dilute the solution to 1 liter and while still hot add 1 to 2 grams of activated carbon, stir and filter through a Buchner funnel. Allow the solution to cool and keep in a warm place. If the solution is allowed to get too cold, some of the guanylurea sulfate will crystallize out. The crystals will redissolve by warming the solution to about 86°F. This reagent may be purchased from the American Cyanamid Company or Eastman Kodak Company.

B. Phenolphthalein Solution: 1 Percent. Dissolve 1.0 gram of phenolphthalein in 50 ml of alcohol (ethyl or isopropyl) and add 50 ml of distilled water.

C. 0.5 Normal Sulfuric Acid Solution. Add cautiously 15 ml of concentrated c.p. sulfuric acid to 1 liter of distilled water.

D. Standard Solution A. Weigh 51.25 grams of c.p. sodium pyrophosphate and dissolve in about 500 ml of distilled water, then dilute to one liter in a volumetric flask.

E. Bromcresol Green Indicator Solution, 1 Percent.

(1) Add 1 gram of bromcresol green powder (tetrabromo-m-cresol-sulfonphthalein) to 50 ml of ethyl alcohol and add 50 ml of distilled water.

(2) Add dilute sodium hydroxide solution from a burette, drop by drop, until all of the bromcresol green powder has dissolved to form a dark blue solution.

F. Zinc Sulfate Solution, 125 g/l.

(1) Dissolve 125 grams of c.p. zinc sulfate in 750 ml of distilled water and then dilute to 1 liter.

(2) Add one ml of 1 percent bromcresol green indicator solution and adjust with dilute sulfuric acid or sodium hydroxide to the yellow end point.

G. Standard 0.3 Normal Sodium Hydroxide Solution. Dissolve 13 grams of c.p. sodium hydroxide in about 500 ml of distilled water, and dilute to one liter. Standardize as follows:

(1) Pipette 50 ml of standard solution A into a 500 ml Erlenmeyer flask and follow steps (g) and (i) as outlined under the procedure for pyrophosphate.

(2) Calculate the pyrophosphate factor F of the sodium hydroxide as follows:

$$\frac{33.4}{\text{ml of standard NaOH}} = \text{factor F.}$$

4.7.3 Ammonia - Proceed as follows:

(a) Assemble an ammonia distillation apparatus as follows.

Place a 500-ml Erlenmeyer flask on an asbestos gauze-covered tripod, closing the flask with a one-hole rubber stopper. Insert in the stopper a length of pyrex glass tubing attached to a vapor trap. At the other end of the vapor trap attach another length of glass tubing; this tubing should also be attached to a water-cooled Liebig or bulb condenser. The delivery tip of the condenser should be inserted into a 500-ml Erlenmeyer receiving flask about 1/4 inch from the bottom. The whole apparatus should be adequately supported by ring stands and clamps and a Bunsen or Tirrill burner used for heating.

(b) Measure exactly 25 ml of standard sulfuric acid solution into the 500 ml receiving flask and dilute to about 150 ml with distilled water.

(c) Add 4 to 5 drops of methyl red indicator solution to the acid.

(d) Place the 500-ml receiving flask under the condenser so that the tip extends about 1/4 inch from the bottom.

(e) Pipette a 10-ml sample of the plating bath into the 500-ml Erlenmeyer flask which is to be used as the distillation flask and dilute to 125 ml with distilled water.

(f) Add about 3 grams of powdered pumice and 5 grams of pellet sodium hydroxide, plus 4 or 5 glass beads, to the 500-ml distillation flask.

(g) Immediately after the addition of the pellets connect the apparatus and place the burner under the flask to ensure that the cold water is running through the condenser.

(h) Continue the distillation until approximately 100 ml of liquid has been distilled over (20 minutes to 1/2 hour).

(i) Remove the 500-ml receiving flask and turn off the flame.

(j) Wash the receiving tip of the condenser with distilled water and collect the washings in the 500-ml Erlenmeyer Flask.

(k) Titrate the solution in the receiving flask with standard sodium hydroxide solution until the color changes from pink to yellow.

(l) Calculate oz/gal of ammonia present according to the following equation.

$$\begin{aligned} & (25 \times N. \text{ of } H_2SO_4) - (ml \text{ of } NaOH) \times (N. \text{ of } NaOH) \times 0.23 \\ & = \text{oz/gal of ammonia (NH}_3\text{)} \end{aligned}$$

4.7.3.1 Preparation of (approximately 0.1 Normal) Sulfuric Acid and 0.1 Normal Sodium Hydroxide Solutions

A. Standard 0.1 Normal Sulfuric Acid (H_2SO_4) - Add 3 ml of concentrated sulfuric acid to about 500 ml of distilled water and dilute to a liter. Standardize the sulfuric acid as follows:

(a) On an analytical balance weigh about 0.2 gram of A. C. S. grade anhydrous sodium carbonate which has previously been dried at 230°F for several hours.

(b) Transfer the carbonate to 250-ml Erlenmeyer flask, add 100 to 125 ml of water and swirl until the carbonate dissolves.

(c) Add 2 to 3 drops of 0.1 percent methyl red indicator solution.

(d) Fill a 50-ml burette with the standard sulfuric acid and titrate until the color changes from yellow to light pink.

(e) Calculate the normality as follows:

$$\frac{\text{Weight of Na}_2\text{CO}_3}{0.053 \times \text{ml of H}_2\text{SO}_4} = \text{Normality of the standard sulfuric acid.}$$

B. Standard 0.1 Normal Sodium Hydroxide:

(a) Pipette 25 ml of the standard 0.1 Normal Sulfuric acid solution into a 250-ml Erlenmeyer flask.

(b) Add 125 ml of distilled water.

(c) Add 2 to 3 drops of 0.1 percent methyl red indicator solution.

(d) Fill a 50-ml burette with the standard sodium hydroxide and titrate until the color changes from pink to yellow.

(e) Calculate the normality as follows:

$$\frac{25 \times \text{N. of H}_2\text{SO}_4}{\text{ml of NaOH}} = \text{Normality of the standard sodium hydroxide}$$

4.7.4 Orthophosphate - Proceed as follows:

(a) Pipette a 3-ml sample of the copper solution into a 400-ml beaker, dilute to 200 ml with distilled water, and add 5 ml of sulfuric acid.

(b) Boil the solution until only 25 ml remains and then cool.

(c) Dilute to 100 ml and add 5 to 10 drops of 1 percent phenolphthalein indicator solution.

(d) Add concentrated ammonium hydroxide slowly until the color changes to pink.

(e) Slowly add concentrated hydrochloric acid drop by drop until the pink color disappears and then add 10 ml in excess.

(f) Add 5 grams of ammonium acetate.

(g) Add 50 ml of "magnesia mixture."

(h) Stir the solution and heat almost to boiling.

(i) Slowly add concentrated ammonium hydroxide until the solution becomes a pink color.

(j) Stir the solution until the precipitate which first forms becomes finely crystalline in structure, (2 to 4 min.).

(k) Cool to room temperature and add concentrated ammonium hydroxide equivalent to 1/5 of the solution volume.

(l) Allow the solution to stand at least 4 hours or overnight.

(m) Filter the residue through a previously ignited and weighed filter or Gooch crucible.

(n) Wash 5 to 10 times with cold 1:1 ammonium hydroxide water.

(o) Moisten the precipitate with a saturated water solution of ammonium nitrate.

(p) Dry the residue slowly either in an oven or inside of another platinum or nickel crucible.

(q) Finally ignite in a muffle furnace at 1100 degrees F, or at a red heat over a Mekker burner for 20 to 30 minutes.

(r) Allow the crucible to cool and reweigh.

(s) Obtain the weight of the precipitate by difference and calculate the oz/gal of orthophosphate according to the following equation:

$$\begin{aligned} & (\text{Weight of ppt} \times 37.9) - [(\text{oz/gal P}_2\text{O}_7) 1.09] \\ & = \text{oz/gal orthophosphate (PO}_4\text{)} \end{aligned}$$

4.7.4.1 Preparation of "magnesia mixture" - Dissolve 55 grams of magnesium chloride (NH_4Cl) in about 800 ml of distilled water. Add 2 ml of concentrated hydrochloric acid and dilute to 1 liter with distilled water.

4.7.4.2 pH Determination - The pH of copper pyrophosphate solutions can be determined by a standard commercial colorimetric comparator set or an electrometric pH meter.

4.8 Copper Fluoborate Solutions

4.8.1 Copper - Proceed as follows:

- (a) Pipette a 5 ml sample into a 250 ml beaker.
- (b) Add ammonium hydroxide until the solution is a deep-blue color.
- (c) Boil gently covered with watch glass until odor of ammonia is faint.
- (d) Add glacial acetic acid until solutions is acid, add one ml in excess and boil for one minute.
- (e) Cool the solution, add 3 to 5 gram of potassium iodide and let stand for 5 minutes.
- (f) Titrate with standard sodium thiosulfate solution and when straw yellow add 5 ml of a 0.1 percent starch solution and continue the titration to disappearance of blue color.
- (g) Read the burette. This value multiplied by the factor for the standard sodium thiosulfate solution gives the ounces of copper per gallon of the plating bath.

Calculations:

$$\frac{\text{Weight copper in 10 ml aliquot} \times 200}{\text{ml of Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O} \times 7.5}$$

Factor in oz/gal for metallic copper. Oz/gal of metallic copper
 $\times 3.72 = \text{oz/gal of copper fluoborate } [\text{Cu}(\text{BF}_4)_2] \text{ in bath.}$

4.8.2 Free Fluoboric Acid - Proceed as follows

(a) Transfer a 10 ml sample to a 250 ml beaker and add 3 drops of cresol red indicator (dissolve 0.10 gram of cresol red in 20 ml of absolute methanol and dilute to 100 ml with distilled water). The 60 gram copper per liter bath is titrated directly and the 120 grams copper per liter bath is diluted with 50 ml water.

(b) Titrate with standard sodium hydroxide solution until the violet blue changes to a greenish blue.

(c) Read the burette. The value multiplied by the factor for the standard sodium hydroxide gives the ounces of fluoboric acid per gallon of the plating bath.

Calculation:

$$\frac{\text{Weight of KHC}_8\text{H}_4\text{O}_4 \times 0.43 \times 100}{\text{ml of NaOH} \times 7.5} = \text{Factor in oz/gal for free fluoboric acid (HBF}_4\text{)}$$

4.8.2.1 pH Determination - The 60-gram copper per liter bath may be controlled by colorimetric pH papers. Readings with pH meters with the glass electrode are unreliable since the pH is affected by the fluoborate ion.

5. PREPARATION FOR DELIVERY

Not applicable to this specification.

6. NOTES

6.1 Intended Use - This manufacturing specification developed by the PE Laboratory of George C. Marshall Space Flight Center is intended for use when copper plating is performed on space launch vehicle components.

6.2 Safety Precautions

6.2.1 Personal Safety - All necessary safety precautions regarding toxicity and industrial health hazards shall be taken by plating personnel under supervision of the Plating Foreman and Safety Engineer in accordance with MSFC instructions and regulations. Adequate ventilation shall be maintained over the plating tanks and proper precautions taken to assure safe conditions in case of spillage. Suitable gloves, protective clothes and devices shall be available.

6.2.2 Chemical Storage - Plating chemicals shall be stored in secure cabinets or chemical storage rooms which can be maintained in a clean and orderly condition, and which can be easily flushed with water to wash away or neutralize spillage.

6.2.3 Handling Containers - All containers employed in the transfer of chemicals shall be washed and cleaned thoroughly immediately after being used.

NOTICE - When Government drawings, specifications or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

TABLE 4-24. PROCESSING MAGNESIUM ALLOYS

Cleaning of Magnesium Alloys	
Sodium carbonate ($\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$)	3 oz/gal
Sodium hydroxide (NaOH)	2 oz/gal
(0.1 oz/gal of soap or synthetic detergent may be added to this solution, if required to remove heavy films of mineral oil.)	
Temperature:	190° to 212°F
Time:	3 to 10 minutes
Current density:	10 to 40 amps/sq ft (cathodic process)
Volts:	6
Pickling of Magnesium Alloys	
Chromic Acid (CrO_3):	24 oz/gal
Temperature:	190 to 212°F
Time:	2 to 5 minutes
Activating of Magnesium Alloys	
Phosphoric Acid (85 percent H_3PO_4)	1.6 pints/gal
Sodium, ammonium potassium	
Acid fluoride:	13-1/3 oz/gal
Temperature:	70 to 90°F
Time:	0.5 to 2 minutes

TABLE 4-25. SOLUTION FOR ZINC IMMERSION
OF ALUMINUM ALLOYS

Zinc Oxide	13 oz/gal
Sodium Hydroxide	70 oz/gal
Temperature	60° to 80°F
Time	30° to 60 seconds

TABLE 4-26. SOLUTION FOR ZINC IMMERSION
OF MAGNESIUM ALLOYS

Zinc Sulfate ($\text{ZnSO} \cdot \text{H}_2\text{O}$)	4 oz/gal
Tetrasodium Pyrophosphate	16 oz/gal
Potassium Fluoride	0.94 oz/gal
Sodium Carbonate	0.67 oz/gal
pH (measured colorimetrically)	10.2 to 10.4
Temperature	175° to 185°F
Time	
Aluminum containing alloys	5 to 7 minutes
Aluminum - Free alloys	3 to 5 minutes
Unalloyed magnesium	3 to 5 minutes
Agitation	Mild

TABLE 4-27. SOLUTION FOR ANODIC ETCH OF TITANIUM

1. Anodic etch in solution containing 79 percent by weight ethylene glycol, 15 percent by weight hydrofluoric acid (as 100 percent) and balance water at 130° to 140°F using at 50 asf for 15-30 minutes.
2. Rinse
3. Copper strike under the following conditions.
 Copper cyanide, 3.1 oz/gal
 Total sodium cyanide, 4.5 oz/gal
 Sodium carbonate (Na_2CO_3), 2.0 oz/gal
 Room temperature; No agitation.
 50 asf for a few seconds, then
 15 asf for 3 to 5 minutes.
4. Rinse

TABLE 4-28. COPPER STRIKE BATHS

	Aluminum Bath 1	Magnesium Bath 2
Copper cyanide, oz/gal	5.5	5.5
Sodium cyanide, oz/gal	6.5	6.8
Sodium carbonate, oz/gal	4.0	4.0
Rochelle salt	8.0	6.0
By analysis		
Copper, oz/gal	3.9	3.9
Free sodium cyanide, oz/gal	0.5	0.75
Conditions		
pH, colorimetric	10.2 to 10.5	9.6 to 10.4
Temperature, °F	100 to 130	150 to 160

TABLE 4-29. ACID COPPER SULFATE BATH

Composition	
Copper Sulfate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, oz/gal	26 to 33
Sulfuric acid, oz/gal	4 to 10
By analysis:	
Copper, oz/gal	5.2 to 6.6
Sulfuric Acid, oz/gal	4 to 10
Conditions:	
Temperature, °F	70 to 120
Current density, asf	20 to 100
Agitation, cathode and/or air	Preferred
Cathode efficiency, percent	95 to 100
Ratio anode to cathode area	1:1
Anodes	Copper
Filtration - continuous preferred, especially for heavy deposits. Voltage - Less than 6 volts generally (Higher for some applications.) Applications - To all ferrous base metals over copper strike.	

TABLE 4-30. COPPER-PYROPHOSPHATE BATH

Composition	
Copper-pyrophosphate, oz/gal	46
Proprietary compound	
Potassium hydroxide, oz/gal	2.4
Ammonia (29 percent), Fl. oz/gal	1.2
By analysis	
Copper, oz/gal	3.0
Pyrophosphate, oz/gal	21.0
Ammonia, oz/gal	0.25
Ratio P_2O_7/Cu	7.0
Conditions	
pH, electrometric	8.8 to 8.2
Temperature, °F	110 to 140
Current density, asf	10 to 75
Voltage, volts	2 to 5 at tank
Agitation, cathode and/or air	
Cathode efficiency, percent	95 to 100
Ratio anode to cathode	1:1 to 2:1
Area	
Anodes	Copper
Filtration	Continuous
Use	Undercoat for bright nickel and chromium
Applications	To all basis metal over copper strike

TABLE 4-31. COPPER - FLUOBORATE BATHS

Composition	Bath 1	Bath 2
Copper fluoborate, oz/gal	30	60
$\text{Cu}(\text{BF}_4)_2$		
Fluoboric acid, HBF_4	to pH	to pH
By analysis:		
Copper, oz/gal	8	16
Sp. gr., ° Be at 80°F	21 to 22	37.5 to 39
Conditions		
pH, colorimetric	0.8 to 1.7	< 0.6
Temperature, °F	80 to 170	80 to 170
Current density, asf	75 to 125	125 to 350
Agitation, cathode preferred, also air		
cathode efficiency, percent	95 to 100	95 to 100
Ratio anode to cathode	1:1	1:1
Area		
Anodes		
Filtration	Copper as required	Copper as required
Volts	6	6-12
Use - Heavy copper deposits to any required thickness. Applications: To all base metals over prior copper strike.		

TABLE 4-32. SAMPLING FOR NONDESTRUCTIVE TESTS

Items In Inspection Lot (Number)	Items in Sample (Percent of Inspection Lot)		Acceptance Number (maximum allowable number of items nonconforming)
	Visual	Thickness	
1 to 6	100	100	0
7 to 15	100	100	0
16 to 40	100	100	0
41 to 110	100	60	0
111 to 300	60	40	1
301 to 500	30	20	1
501 and over	20	15	2

TABLE 4-33. SAMPLING FOR DESTRUCTIVE TESTS

Items In Inspection Lot (Number)	Items In Sample (Number)	Acceptance Number (maximum allowable number of items nonconforming)
1	0	0
2 to 5	1	0
6 to 25	3	0
26 to 100	4	0
101 to 300	6	0
301 to 5000	10	0
5001 and over	15	0

TABLE 4-34. SPECIFIC GRAVITY OF COPPER SULFATE SOLUTIONS

Specific Gravity 77/39.2°F	° Be (77°F)	Total Copper Sulfate Plus Sulfuric Acid (oz/gal)
1.01	1.44	2.7
1.02	2.84	4.8
1.03	4.22	7.0
1.04	5.58	9.1
1.05	6.90	11.3
1.06	8.21	13.4
1.07	9.50	15.7
1.08	10.07	17.8
1.09	12.00	20.0
1.10	13.00	22.3
1.11	14.40	24.5
1.12	15.50	26.8
1.13	16.70	29.1
1.14	17.80	31.3
1.15	18.90	33.6
1.16	20.00	35.9
1.17	21.10	38.3
1.18	22.10	40.6
1.19	23.20	43.0
1.20	24.20	45.4
1.21	25.20	47.8
1.22	26.20	50.2
1.23	27.10	52.6

MANUFACTURING SPECIFICATION FOR
THE USE OF MARKING INKS ON ALUMINUM SURFACES

1. SCOPE

1.1 Scope - This manufacturing specification covers the approved PE Laboratory requirements for the use of marking inks on bare, clad, or unclad aluminum alloy surfaces.

1.2 Applicability - The requirements set forth in this specification cover the application and the removal of marking inks to and from types 2014, 2219, 5086, 5456, 6061, and 7075 bare, clad, or unclad aluminum alloys.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this specification, form a part of this specification.

SPECIFICATIONS

Federal

0-A-51C

Acetone, Technical

STANDARDS

Federal

141A

Paint, Varnish, Lacquer, and
Related Materials; Methods of
Inspection, Sampling and Testing.

2.2 Other Publications - The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

Speedry Products, Inc.

Form S-426

Magic Marker

(Copies of this publication may be obtained from Speedry Products, Inc., 121st St. and Atlantic Avenue, Richmond Hill, L.I., N.Y.)

3. REQUIREMENTS

3.1 General - No deviation from the requirements of this manufacturing specification shall be permitted without prior written approval of the MSFC initiating activity.

3.2 Materials

3.2.1 Marking ink - The ink used for marking shall be equal or equivalent to the commercial product known as "Magic Marker." It shall be air drying, permit usage from absorbent pads, be nonetching, be nontoxic, and shall dry by evaporation within 5 minutes after application at a temperature of 73.5° F ($\pm 2^\circ$) and a relative humidity of 65(± 2) percent. Furthermore, the ink film which has dried on 0.020-inch thick aluminum alloy sheet shall possess sufficient plasticity to permit forming over a round bar 1 inch in diameter without peeling.

3.2.2 Base metal - The metallic surfaces to which marking ink is to be applied shall be dust-free and of a cleanliness equivalent to sheet aluminum received from the mill in the dry, uncoiled condition.

3.3 Application - The marking ink may be applied by stamp, brush, roller, or air brush as may be required.

3.4 Marking Ink Removal - The ink used shall be easily removed by swabbing with acetone or other recommended solvent.

3.5 Limitations - Marking inks shall not be applied to the following kinds of aluminum alloy surfaces:

- Anodized or conversion coated surfaces.
- Surfaces which are to be primed without a prior cleaning operation.
- Surfaces which have already been primed and are to be painted.
- Surfaces which have been LOX cleaned and might subsequently come in contact with either liquid oxygen or hydrogen peroxide.

4. QUALITY ASSURANCE PROVISIONS

4.1 General - When the requirements of paragraph 3.2.1 are met, marking inks shall be judged primarily by the sharpness of the registration that they produce.

4.2 Testing Procedures - As a guide towards evaluating the characteristics of marking inks the procedures in Standard 141A shall be used.

5. PREPARATION FOR DELIVERY

Not applicable to this specification.

6. NOTES

6.1 Intended Use - This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use when marking aluminum alloy surfaces for identification purposes.

6.2 Safety Precautions - All necessary precautions regarding toxicity and fire hazards shall be taken in accordance with MSFC instructions and regulations.

6.3 Storage - Marking inks, because of the volatility of the thinner used as a vehicle for pigments, shall not be stored in quantities greater than required for a 90-day consumption. They shall not show any settling, curdling, livering, or skins when stored for periods of up to 90 days.

6.4 Definitions

6.4.1 Curdling - The tendency of an ink to congeal or thicken into a liquid too viscous for use as intended.

6.4.2 Livering - The coagulation of an ink into a viscous, rubberlike mass.

6.4.3 Settling - The decrease or loss in the dispersed, solid, colored phase of an ink due to the formation of either soft or solidified deposits on the bottom of the ink container.

6.4.4 Skinning - The tendency of an ink to form a surface layer or skin. This tendency is accelerated in an open or partially filled container.

NOTICE: When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government

procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING PROCESS FOR THE CLEANING OF FLARED AND UNFLARED TUBING

1. SCOPE

1.1 Scope - This process covers the Process Engineering Laboratory requirements for the cleaning of flared or unflared aluminum or stainless steel tubing used in LOX, fuel, and pneumatic systems of space launch vehicles.

1.2 Applicability - Cleaning procedures outlined in this process are applicable to flared or unflared tubing made of the following materials:

- Stainless steel - Grades 304, 304L, 316, 321, and 347.
- Aluminum - Alloys 5052, 5086, 5456, and 6061.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this process, form a part of this process.

SPECIFICATIONS

Federal

0-H-795(2)	Hydrofluoric Acid, Technical
0-N-350	Nitric Acid, Technical
0-0-670	Orthophosphoric (Phosphoric) Acid, Technical
0-P-94a	Paper, Test, pH Indicator
0-S-595a(3)	Sodium Dichromate, Technical-Grade (Sodium Bichromate) Sodium Dichromate Dihydrate
0-S-598	Sodium Hydroxide, Technical

0-S-642B	Sodium Phosphate, Tribasic, Technical, Anhydrous, Dodecahydrate and Monohydrate
0-T-634a	Trichloroethylene, Technical
BB-N-411a	Nitrogen
UU-T-81f(1)	Tags, Shipping and Stock

Military

MIL-A-00148C(2)	Aluminum Foil
MIL-A-6091B	Alcohol, Ethyl, Specially Denatured, Aircraft

George C. Marshall Space Flight Center

MSFC-SPEC-164	Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic Systems, Specification for
MS 101.0	Manufacturing Specification for The Drying of Components in a Vacuum

3. REQUIREMENTS

3.1 General - No deviation from the requirements of this manufacturing process shall be permitted without prior written approval of the Process Engineering Laboratory.

3.2 Materials

3.2.1 Liquid solvent - Trichloroethylene used for liquid or vapor degreasing shall be equivalent to Type II of Specification 0-T-634a.

3.2.2 Test solvent - Trichloroethylene used for testing the cleanliness of sample tubing shall meet the requirements for Type I of Specification 0-T-634a with the exception that the nonvolatile residue shall not be greater than 0.010 gram per 500 cc when the solution is evaporated at 221° to 230° F and that no particle larger than 175 microns in any dimension shall be present.

3.2.3 Demineralized water - Demineralized water shall contain no particle larger than 175 microns in any dimension and shall possess a minimum resistivity of 50 000 ohms.

3.2.4 pH indicator - The pH indicating paper used in this process for checking neutrality shall be of the Medium Range, Class A, of Specification 0-P-94a.

3.2.5 Aluminum foil - Dry annealed aluminum foil shall conform to paragraph 6.3.2 of Specification MIL-A-00148C.

3.2.6 Tags - Tags employed in marking of cleaned components shall conform to Style C of Specification UU-T-81f(1).

3.2.7 Drying and preservation gases - The nitrogen or air employed for drying or preservation shall be filtered to a 100 micron level, shall possess an oil content not greater than 3 parts per million by weight, and shall contain a maximum of 24 parts per million by volume of moisture. Samples of gas shall be taken upon receipt of each bottle of gas for compliance to the applicable specification.

3.2.8 Compressed, heated gases - When drying is performed by blowing or purging a component with heated, compressed gases, the pressure shall be 15 to 25 psi and the temperature shall be $180^{\circ} \pm 20^{\circ}$ F. The gas shall conform to paragraph 3.2.7 of this process.

3.3 Cleaning Procedures

3.3.1 Flared stainless steel tubing (internal surfaces only)

3.3.1.1 Degrease - Circulate liquid trichloroethylene through the tubing for 3 to 5 minutes at ambient temperature and at a pump pressure of 30 ± 10 psi. Note safety precautions given in paragraph 6.2 of this process.

3.3.1.2 Dry - Using compressed air (see paragraph 3.2.7) remove all traces of trichloroethylene by drying at $180^{\circ} \pm 20^{\circ}$ F for 1 to 2 minutes at 15 to 25 psi.

3.3.1.3 Hot alkaline wash - Circulate a solution of 10 ± 2.0 percent by weight of trisodium phosphate through the tubing at $180^{\circ} \pm 10^{\circ}$ F for 10 to 15 minutes.

3.3.1.4 Rinse - Circulate tap water at a temperature of 160 to 180° F through the tubing for 5 to 10 minutes.

3.3.1.5 Pickling solution - Pickling solution is to be used at this point, if required, for internally corroded or contaminated tubing only. Do not use pickling solution if the tubing has been previously cleaned by pickling, bright annealing, or other equivalent etchant processes. Protect the flared part of the tubing while circulating through the tubing for 5 to 8 minutes the following aqueous solution at a temperature of 90° to 100° F. (Sodium bifluoride may be used with excess nitric acid to produce the required hydrofluoric acid.)

20 ± 2 percent (by weight) nitric acid
1.5 ± 0.5 percent (by weight) hydrofluoric acid

3.3.1.6 Rinse - Circulate tap water through the tubing at a temperature of 160° to 180° F for 5 to 10 minutes.

3.3.1.7 Passivate - Passivate the tubing by circulating through the tubing for 15 to 20 minutes at 125° ± 5° F the following aqueous solution:

20 ± 2 percent (by weight) nitric acid
2.0 ± 0.5 percent (by weight) sodium dichromate

3.3.1.8 Rinse - Circulate demineralized water through the tubing at ambient temperature until the pH of the effluent is 6 to 8.

3.3.1.9 Pressure-dry - Using nitrogen or air at 180° ± 20° F dry by flushing gas through the interior of the tubing for 15 to 20 minutes at 15 to 25 psi.

3.3.1.10 Vacuum dry - When applicable, vacuum dry in the manner outlined in Specification MS 101.0.

3.3.2 Unflared stainless steel tubing

3.3.2.1 After lubrication with oil

A. Vapor degrease in the manner outlined in Specification MS 150.0A. Flush the internal surfaces of the tubing with liquid trichloroethylene by using the pump on the vapor degreaser.

B. Using air that meets the requirement of paragraph 3.2.8, remove all traces of trichloroethylene by drying at 180° ± 20° F for 1 to 2 minutes at 15 to 25 psi.

3.3.2.2 After lubrication with soap

A. Flush with tap water at 160° to 180° F until free of soap.

NOTE: If hot tap water does not remove the soap, use an aqueous solution containing $5 (\pm 2)$ percent by weight of sodium hydroxide at 160° to 180° F.

B. Rinse by flushing with demineralized water at 160° to 180° F for a minimum of 5 minutes.

C. Dry by flushing nitrogen or air through the inside of the tubing at $180^{\circ} \pm 20^{\circ}$ F for 15 to 20 minutes at 15 to 25 psi.

3.3.3 Aluminum tubing - Prior to cleaning flared aluminum tubing, protect the flared ends of the tubing by connecting sections of tubing together with aluminum AN fittings which fit into the tube flare and protect the flare surfaces from contact with the chemical cleaning solution.

3.3.3.1 Degrease - Circulate liquid trichloroethylene through the tubing for 3 to 5 minutes at ambient temperature and at a pump pressure of 30 ± 10 psi. Note the safety precautions given in paragraph 6.2.

3.3.3.2 Dry - Using compressed air, remove all traces of trichloroethylene by drying at $180^{\circ} \pm 20^{\circ}$ F for 10 to 20 minutes at a pressure of 15 to 25 psi.

3.3.3.3 Hot alkaline degrease - Circulate for 5 to 10 minutes a solution of 10 ± 2.0 percent by weight trisodium phosphate through the tubing. Maintain a temperature of 170° to 190° F and a pressure of 40 ± 20 psi on the circulating solution.

3.3.3.4 Rinse - Circulate tap water at ambient temperature through the tube for 5 to 10 minutes.

3.3.3.5 Nitric acid rinse - Circulate through the tube an aqueous solution of 5.0 ± 1 percent by weight of nitric acid at 85° to 100° F for 5 to 8 minutes.

Alternate phosphoric acid rinse - An aqueous solution of 5.0 ± 0.5 percent by weight phosphoric acid may be used to remove heavy soils that cannot be removed by the nitric acid rinse in paragraph 3.3.3.5 above. In these cases, circulate the solution through the tubing at 160° to 180° F for 5 to 8 minutes.

3.3.3.6 Cold rinse - Circulate and rinse with demineralized water at ambient temperature until the pH of the effluent is between 6 and 8.

3.3.3.7 Dry - Dry per paragraph 3.3.1.9 of this process.

3.3.3.8 Vacuum dry - When applicable, vacuum dry per paragraph 3.3.1.10 of this process.

3.4 Recleaning Procedure - If additional cleaning, or recleaning, of stainless steel tubing (paragraphs 3.3.1 and 3.3.2) or aluminum tubing (paragraph 3.3.3) becomes necessary, the tubing must be cleaned to the original cleanliness level by flushing with trichloroethylene which has been filtered to remove all particles larger than 100 microns (absolute) in any dimension. Make particle count and stop cleaning when the requirements of Specification MSFC-SPEC-164 have been met. Repeat the operation if necessary to meet these requirements.

4. ACCEPTABILITY PROVISIONS

4.1 Cleanliness - Tubing cleaned by the procedures outlined in this process must meet the requirements of paragraphs 3.5.1.1, 3.5.1.2, 3.5.2.1, 3.5.2.2, and the qualification procedures of paragraph 4.2.2.3 of Specification MSFC-SPEC-164.

4.2 Quality Control - Control of the quality of various tank solutions will be assured by adherence to the times, temperatures, and concentrations specified.

5. PREPARATION FOR DELIVERY

5.1 Preservation and Packaging - Tubing shall be sealed with PE Laboratory approved coverings or closures. The sealed tubes shall be placed in clean polyethylene bags purged by nitrogen or air meeting the requirements of paragraph 3.2.7 of this process. The bag openings shall be heat sealed to prevent contamination of the tubes. (Refer to the applicable section of Saturn Packaging Manual.)

5.2 Marking - Tubing cleaned by the procedures outlined in this process shall be marked with tags conforming to Specification UU-T-81f(1), Type C, which shall bear the following information.

- Part or identification number.
- Method of cleaning and micron level.
- Date of cleaning.
- Title and date of this process.
- Any further information required by contract.

6. NOTES

6.1 Intended Use - This manufacturing process, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use in the cleaning of aluminum or stainless steel tubing used in LOX, fuel and pneumatic systems.

6.2 Volatile Solvent Safety Precautions - Perform this operation under fume exhaust hood or use room exhaust system. Use gloves and chemical respirator if no exhaust system is available.

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING PROCESS DATA FOR
CLEANING OF ALUMINUM TUBING ENDS
PRIOR TO FLARING OPERATIONS

1. SCOPE

1.1 This manufacturing process data cover the approved PE Laboratory requirements for etch cleaning of 6061 aluminum tube ends prior to flaring of the tube ends.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Tube handling devices
- Pyrex beaker
- Hot plate
- Pyrex rinsing beaker
- Rubber gloves
- Safety goggles

2.2 Materials

- Sulfuric acid (40 ± 5 percent by wt)
- Sodium dichromate (5 ± 1 percent by wt)
- Deionized water
- Missile grade air
- Litmus paper
- Trichloroethylene
- Aclar or Polyethylene

3. PROCEDURE

3.1 Prepare chemical etch solution by adding sulfuric acid (40 ± 5 percent by wt) to sodium dichromate (5 ± 1 percent by wt) in a pyrex beaker with an amount as determined by the size of tubing end to be subsequently flared.

3.2 Remove any excess lubricant, stains, and heavy concentration of contamination from tubing ends by vapor degreasing.

3.3 Allow the tubing end to statically dry.

3.4 Maintain the etch solution as prepared in paragraph 3.1, 'at $145^{\circ} + 5^{\circ} \text{F}$ on a hot plate. Immerse the tubing end 1 inch minimum distance in this heated solution for $4 (+ 1)$ minutes.

CAUTION: Hold tubing end vertically, being careful not to allow the etch solution to become entrapped in the tubing configuration any further than the amount of the tubing end to be etched, subsequent to being flared.

3.5 Rinse the tubing end in deionized water.

3.6 Dry the tubing end with missile grade air.

4. CONTROL

WARNING: Personnel should exercise all safety regulations as to handling of acids (use rubber gloves to prevent the acid from contacting the skin and goggles to cover the eyes in case of splattering or spillage of acid).

CAUTION: Care should be made to prevent acid fumes from coming in contact with materials which would be deleteriously affected.

4.1 The tube ends after rinsing should have pH within the range of 6.0 to 8.0 as determined with litmus paper.

4.2 After etching, handle the tubing with care to prevent recontamination of the etched surfaces to be flared.

4.3 The etched tube ends will be protected by covering the tube with a clean package material, such as aclar or polyethylene, and taped about 4 to 5 inches from each end of the tube to be flared.

MANUFACTURING SPECIFICATION FOR
THE APPLICATION OF DRY FILM LUBRICANTS

1. SCOPE

1.1 Scope — This manufacturing specification covers the approved PE Laboratory requirements for the application of dry film lubricants and anti-seize compounds, that are compatible for use in LOX or fuel service, to space launch vehicle components.

1.2 Classification — This specification covers the following classes of lubricants —

- Class A — Sprayed on and baked.
- Class B — Brushed on and air dried.
- Class C — Wiped on, paste type.
- Class D — Dry, powder type.

NOTE: Typical components presently utilizing dry lubricants are the following aluminum, steel and non-metallic parts.

- Aluminum "B" nuts and fittings
- Steel "B" nuts and fittings
- Door hinges
- Valve components
- "O" rings

2. APPLICABLE DOCUMENTS

2.1 Governmental — The following documents of the issue in effect on the date of use of this specification form a part of this specification to the extent indicated herein.

SPECIFICATIONS

Federal

UU-T-81D (1)	Tags, Shipping and Stock
PPP-T-60	Tags, Pressure Sensitive Adhesive, Waterproof

Military

MIL-C-5541(1)	Chemical Films for Aluminum and Aluminum Alloys
MIL-M-7866A (ASG)	Molybdenum Disulfide Powder, Lubricant
MIL-A-8265	Anodic Coatings for Aluminum and Aluminum Alloys
MIL-S-15847	Spray guns and Accessories, Paint and Dope, Aircraft Use
MIL-P-16232B, Type M, Class 3	Phosphate Coatings, Heavy, Manganese, or Zinc Base
MIL-P-17667A, Type I	Paper, Wrapping, Chemically Neutral

George C. Marshall Space Flight Center

MSFC-SPEC-106	Compatibility Testing, Liquid Oxygen System Materials, Specification for
MSFC-SPEC-110	Cleanliness of Components for use in Liquid Oxygen, Fuel Pneumatic Systems.

STANDARDS

Military

MIL-S-171	Systems for Preparation, Painting and Finishing Metal and Wood Surfaces
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3. REQUIREMENTS

3.1 General — No deviation from the requirements of this manufacturing specification shall be permitted without prior written approval of the MSFC initiating activity.

3.2 Equipment

3.2.1 General — The equipment used in applying and conditioning dry film lubricants and anti-seize compounds on space launch vehicle components shall be suitable and adequate for producing a coating that will comply with the requirements of this specification.

3.2.2 Spray equipment — Spray equipment shall comply with the requirements of Specification MIL-S-15847. The spray nozzle shall be made of stainless steel or possess a hard-chrome finish. It shall be used for no purpose other than applying Class A, dry film lubricant or anti-seize compound.

3.2.3 Magnetic stirrer — A stirring and mixing device of the magnetic type shall be available for compounding or for keeping molybdenum disulphide in suspension.

3.2.4 Gloves — Gloves used by personnel applying dry film lubricants or handling components shall be polyethylene, or equal, for use in critical service applications and nylon, or equal, for use in noncritical service applications.

3.2.5 Wiping materials — Wiping materials shall be those specified below.

3.2.5.1 Lint-free, dust-free wipers — WA-400; Urethane, foam wipers or equal, as supplied by Shelley Urethane Industries, 4528 Brazil Ave., Los Angeles, California, may be used in the application of lubricants or for removing contaminants and excess lubricant.

3.2.5.2 Paper towels — Dust-free and lint-free, soft paper towels for use in cleaning shall be available.

3.2.5.3 Cloths — Lint-free cloths for use in noncritical applications shall be available.

3.2.6 Masking tape — Tape that will not leave any adhesive on parts when removed shall be used for masking areas not to be lubricated or for the adhesion test on noncritical areas.

3.2.7 Kraft paper — Paper employed for masking shall be a neutral, non-corrosive, kraft-type, conforming to Specification MIL-P-17667, Type I.

3.2.8 Baking oven — An oven capable of heating treated parts to 400° F shall be available. It shall be a self-venting or recirculating oven.

3.2.9 Infrared lamps — Infrared lamps shall be used for baking applied coatings when parts cannot be heated in an oven. Lamps shall be equipped with screen guards and fastened in secured holders.

3.2.10 Vacuum cleaner — Self-contained vacuum equipment of the portable type shall be available in all lubricant application areas. This equipment shall receive regular, periodic servicing and maintenance.

3.3 Facilities

3.3.1 General — The areas where dry film lubricants or anti-seize compounds are applied shall conform to the requirements for the particular application.

3.3.2 Areas, for critical applications — These rooms or areas shall be completely dust free and devoid of any spray painting or work which might create contaminates.

Tables, trays, racks, and other equipment shall be constructed of metal or other hard, impervious material which will not absorb lubricants or compounds.

The humidity and temperature in these rooms shall be controlled. A slight positive pressure of filtered air without undue air currents shall be maintained.

The room shall be provided with vacuum facilities for keeping the immediate working areas free of dirt, lint, and excess lubricating material. The integrated vacuum equipment shall vent to the outside of the room or building.

3.3.3 Areas, for non-critical applications — A clean table or elevated surface located in an area comparatively clean of dirt and contaminants and adjacent to the regular assembly operation will be adequate for the application of dry film to non-critical components. These tables shall be used only for the application of dry-film lubricants, and shall be supplied with all necessary equipment for applying the coatings.

3.4 Materials

3.4.1 Molybdenum disulphide — The lubricants or anti-seize compounds covered in this specification shall be basically molybdenum disulphide which meets as a minimum the requirements of Specification MIL-M-7866A (ASG) and which has a maximum grain size of 1 micron (0.0004 inch). The vehicle, solvent, or carrier must be completely volatile and must leave the coating LOX-insensitive as determined by the liquid oxygen impact test, Specification MSFC-SPEC-106.

3.4.1.1 Class A — An oxygen-safe, dry, film lubricant of sprayable type, that requires baking to remove all traces of impact-sensitive solvent, carrier or vehicle.

3.4.1.2 Class B — An oxygen-safe, dry, film lubricant of the brush-on type that requires only air-drying to obtain the desired coating. It may be used as an anti-seize compound, a corrosion preventative on bare exposed parts in LOX systems, and in areas requiring less stringent lubricating qualities.

3.4.1.3 Class C — An oxygen-safe, dry, film lubricant to be used primarily as an anti-seize compound and that may be wiped on as a paste where the coating thickness is not as critical as in Class A or Class B lubricant service.

3.4.1.4 Class D — An oxygen-safe, micro fine-grained, molybdenum disulphide powder to be applied in dry form on parts requiring a minimum of lubrication. Its quality shall permit the compounding of Classes A, B, and C lubricants.

3.4.1.5 Aerosol or squeeze type — Dry-film lubricants supplied in squeeze bottles or aerosol type pressurized containers are permitted, but they must meet the requirements of the class of lubricant for which they are used. Molyube Spray Lubricant, or equal, manufactured by Bel-Ray Co., Inc., 111 Green Village Road, Madison, N. J., is of this type.

3.5 Procedure

3.5.1 Class A lubricant

3.5.1.1 Degrease — Prior to pretreatment, all parts shall be vapor degreased per Specification MS 100.8 or by other suitable methods that do not cause corrosion or difficulties in further processing.

3.5.1.2 Pretreatment — Metal surfaces shall be pretreated to promote adherence of the lubricant as follows.

A. Aluminum and aluminum alloys

- Anodic coating — Surfaces as specified by engineering drawing shall be anodized per Specification MIL-A-8625A.
- Conversion coating — Chromium-chromate coatings when specified by engineering drawing shall be Iridite 14-2, or equal, which conforms to Specification MIL-C-5541.

B. Carbon and low alloy steels

- Phosphate coating — Degreased, steel surfaces shall be phosphatized using a manganese phosphate base material conforming to Specification MIL-P-16232B, Type M, Class 3.

C. Corrosion resistant steels — The 300 series, 400 series, and precipitation hardening 17-4PH, 17-7PH, and 15-7 Mo shall be blasted using abrasive of 120 to 325 mesh size, either dry or admixed with water. If dimensional tolerances are critical omit the blasting operation.

- Passivate — Passivate in an aqueous solution of 20 percent nitric acid and 2 percent sodium dichromate (by weight) at a temperature of 125° F (+ 5°) per Specification MS 100.4.
- Precision parts — Parts such as valve components, shall not be pretreated. These surfaces shall be lubricated as specified on the applicable drawing.

3.5.1.3 Masking — Tape and a neutral, noncorrosive kraft paper shall be used to mask-off areas not to be lubricated. A minimum use of tape is preferred; however, where the lubricant must be sharply confined, press the tape firmly in position as needed.

3.5.1.4 Application

A. Dipping — Class A lubricant shall be used for dipping small parts that are to be completely coated. They shall be tumbled or spray-tumbled and allowed to air-dry for 30 minutes before baking.

B. Spraying — Spraying shall be done in areas meeting the requirements of paragraph 3.3.2 of this specification. Parts to be sprayed shall be placed on a screen or rack which is free of build-up from previous usage. The lubricant sprayed shall have been stirred by a magnetic stirring device to a uniform consistency. If lumpiness appears, strain the lubricant thru a 100-mesh screen or two thicknesses of lint-free cloth. Return the lubricant to the magnetic stirrer and repeat stirring prior to usage. Should spraying be from aerosol containers, shake the container well before each spraying.

When a spray gun is used, adjust the air pressure to 20 to 30 psi at the gun and adjust the spray for very fine atomization. Hold the gun at a right angle to the surface and apply a wet coating of uniform thickness by spraying in a cross pattern. A final coating thickness of 0.0003 to 0.0007 inch, usually specified on engineering drawings, requires approximately six passes to achieve.

After spraying, return the lubricant cup to the stirrer. If spraying is completed, thoroughly clean the gun and other equipment with water.

C. Drying — Coatings shall be allowed to air-dry thoroughly prior to turning or moving parts or subsequent baking. This provision minimizes marring of coatings. Coatings must not be touched prior to baking and operators must wear polyethylene gloves for handling LOX system parts and nylon gloves for handling non-LOX service parts.

D. De-mask — Remove all masking prior to baking of coatings (Table 4-35).

TABLE 4-35. BAKING SCHEDULE

Material	Temperature (°F)	Time (Hours)
Aluminum alloys, 2014-T6, 6061-T6	326	3.5
Other aluminum alloys	275	1
Steels and steel alloys	400	1

Case hardened steels	300	1
Stainless steels (except type 440-C)	400	.2. 5
Type 440-C, Stainless Steel	300	1.
Titanium	400	1
Plated surface (except as limited by base metal)	400	1

3.5.2 Class B lubricant

3.5.2.1 Degrease — Degrease per paragraph 3.5.1.1 of this specification.

3.5.2.2 Pretreatment — Pretreat the parts per paragraph 3.5.1.2 of this specification.

3.5.2.3 Masking — Mask as outlined in paragraph 3.5.1.3 of this specification.

3.5.2.4 Application — Using a lubricant of uniform consistency and employing a clean brush of the correct size, brush the lubricant onto the desired areas. The operator shall wear approved gloves. The thickness of the wet coating shall be 0.0003 to 0.0007 inch.

3.5.2.5 Air-dry — The coating shall be allowed to air dry completely or for a minimum of 8 hours before assembly.

3.5.2.6 Application techniques — Examples of Class B, lubricant applications are as follows.

A. Fluid line assemblies (B nuts) — Lubricants shall be applied sparingly to the shaded threaded area in Figure 4-24. Care shall be exercised to avoid dripping onto adjacent areas or contaminating surfaces in contact with the medium.

B. Sleeveless assemblies — Flared lines with AN817 couplings shall be coated as shown in Figure 4-25. This lubricant application prevents twisting of the line when the nut is tightened.

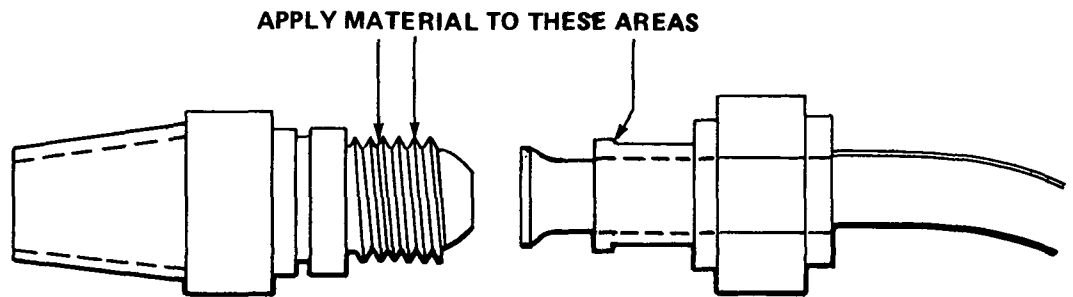


Figure 4-24. Typical fluid line assembly.

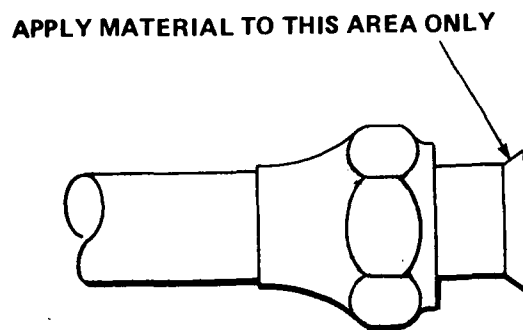


Figure 4-25. AN 817 nut assembly.

C. Flareless assemblies (MS & Ermeto) — Lubricant shall be applied to MS fittings as indicated in Figure 4-26 by the heavy lines.

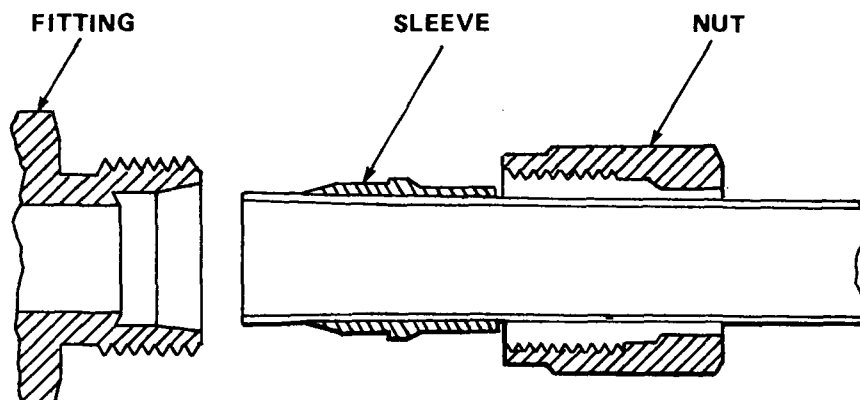


Figure 4-26. Flareless assembly.

D. Tapered pipe threads — Lubricant shall be applied to only male pipe threads as shown in Figure 4-27. Care shall be taken to avoid coating the first two threads. If lubricant contacts the nose, it must be carefully removed by wiping until a clean nose is attained.

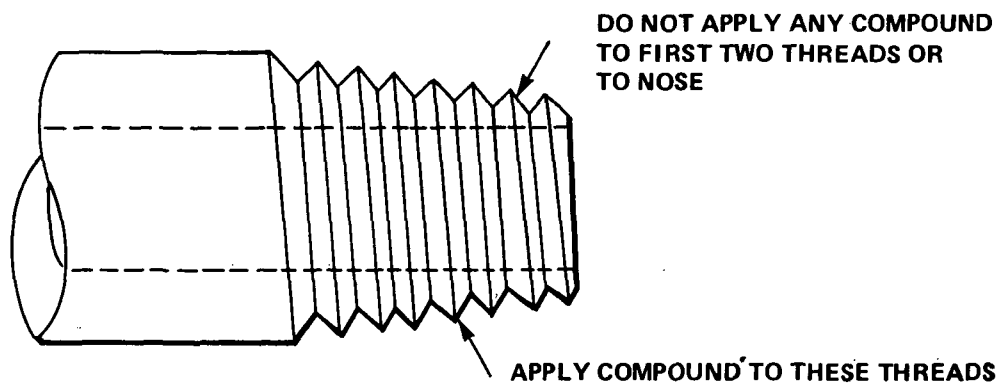


Figure 4-27. Tapered pipe threads.

E. Moving parts — Class B lubricant shall be applied to the entire contact surface of moving parts, such as valve pistons, when such parts require lubrication or corrosion protection.

3.5.2.7 Excess lubricant — Excess lubricant shall be removed from coatings of Class B type by wiping with a water-moistened, approved, lint-free cloth.

3.5.3 Class C lubricant

3.5.3.1 Degrease — Degrease as specified in paragraph 3.5.1.1 of this specification.

3.5.3.2 Pretreatment — Pretreat parts as specified in paragraph 3.5.1.2 of this specification.

3.5.3.3 Masking — Mask as specified in paragraph 3.5.1.3 of this specification.

3.5.3.4 Application — Class C lubricant shall be applied with a clean brush or by wiping with a lint-free cloth and in accordance with the engineering drawing. Excess lubricant shall be wiped off with a water-moistened lint-free wiper.

3.5.4 Class D lubricant

3.5.4.1 Degrease or clean — Metal parts shall be degreased as specified in paragraph 3.5.1.1 of this specification, whereas non-metallic components shall be cleaned as specified in Specification MS 100.9.

3.5.4.2 Pretreatment — If applicable, pretreat surfaces per paragraph 3.5.1.2 of this specification.

3.5.4.3 Masking — Mask per paragraph 3.5.1.3 of this specification.

3.5.4.4 Application — Class D lubricant shall be applied by rubbing parts with a cloth or wiper impregnated with lubricant in order to coat the surface with a very fine layer. The coating shall be burnished with a clean, dry cloth or soft paper towel to produce a smooth coating.

Flat, plastic parts shall be coated by placing lubricant on a flat cloth and by rubbing the part against the cloth.

Rubber parts shall be coated by covering the parts with lubricant in a shallow pan and by rubbing the lubricant against the parts by use of glove-covered fingers. The excess powder shall be removed by vacuum brush. Do not use compressed air to blow the excess powder off the parts.

3.6 Removal of Dry-Film Lubricants

3.6.1 Vapor hone — If it is necessary to remove a coating, vapor-hone or grit blast with 120- to 325-mesh alumina when dimensional tolerances are not critical. If restrictive tolerances do not permit vapor honing, use a chemical means of removing the coating as shown in paragraph 3.6.2.

3.6.2 Stripping solutions

A. No. 1, 15-percent chromic acid (by weight) in water at 160° to 180° F.

B. No. 2, 15-percent sodium hydroxide plus 5-percent sodium carbonate (by weight) in water at 160° to 180° F.

A typical materials and solution schedule is shown in Table 4-36.

TABLE 4-36. TYPICAL MATERIALS AND SOLUTIONS SCHEDULE

Base Material	Solution Number	Remarks
Aluminum bronze	2	Coating does not dissolve. It strips off in sheets.
Aluminum bronze	1	Leave in solution for a minimum time, since it attacks metal slowly.
7075-T6, aluminum, anodized	1	Removes anodizing (Solution No. 2 attacks metal).
4130 steel — 0.0002-inch chromium plate	1 or 2	
Stainless steel	1 or 2	
4130 steel — 0.0002-inch nickel plate	1 or 2	
4130 steel, cadmium plated	1	Removes plating
4130 steel, cadmium plated	2	
4130 steel	1 or 2	

3.6.3 Stripping procedure — The part shall be immersed in the solution a minimum length of time to remove the coating. After stripping, the part shall be thoroughly washed in water and before recrating shall be pretreated per Table 4-36 of this specification.

3.7 Repair of Dry-Film Lubricant — When the dry-film, lubricant coating is worn or in need of repair, clean parts with a LOX insensitive solvent, abrade the film at right angles with emery paper of 180- to 240-grit, reclean with solvent, and, finally, spray, brush, or wipe lubricates the defective area.

4. QUALITY ASSURANCE PROVISIONS

4.1 General — To prevent misapplication of dry-film lubricants, only LOX-insensitive lubricant shall be applied.

4.1.1 Acceptability — The acceptability of dry-film lubricants shall be used on paragraph 4.4.5 of Specification MIL-M-7866A(ASG) as a minimum requirement plus such other qualifying provisions imposed by MSFC procuring activity.

4.1.2 Cleanliness — Parts shall be inspected for cleanliness prior to the application of dry-film lubricants.

In the case of LOX components, the requirements of paragraph 4.4.1.1 of Specification MSFC-SPEC-110 for surfaces in contact with the medium shall be maintained.

4.1.3 Appearance — The lubricant coating shall be uniformly — gray to black in color. It shall be smooth and free from blisters, irregularities, scratches, surface imperfections, and it shall show no separation of ingredients.

NOTE: Slight discoloration of stainless steel parts is acceptable.

4.1.4 Thickness — The maximum, permissible thickness of a sprayed or brushed coat shall be 0.001 inch, unless otherwise specified by engineering drawings.

4.1.5 Adhesion — For LOX components, the fingernail test shall suffice. Do not employ the tape test.

The tape test shall apply on non-LOX components. It shall consist of pressing a short length of tape onto a surface and subsequently removing it abruptly with one stroke. No lifting or peeling of the coating is permitted.

4.1.6 Cleanliness of equipment — All means of applying dry-film lubricants shall be clean and suitable for the application. Meticulous care must be exercised to clean utensils, cups, mixer, spray-gun, and brushes with water after every day's operation.

5. PREPARATION FOR DELIVERY

5.1 Preservation and Packaging — Components which have been lubricated shall be protected from contamination and damage by placing in sealed, polyethylene bags and packed as may be necessary to prevent damage. Difficult to package items shall be protected only where protection is necessary.

LOX components shall be packaged and preserved as required by Specification MSFC-SPEC-110.

5.2 Marking — Lubricated and packaged parts shall be tagged. The tags shall bear the following information.

- Type of dry-film lubricant
- Intended use of the part (if LOX service, be specific),
- All information required by contract

6. NOTES

6.1 Intended Use — This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended to specify the requirements of the application of dry film lubricants and anti-seize compounds for use in space launch vehicle in areas where LOX and fuel service compatibility is needed.

6.2 Baking Precautions — Where sealants, adhesives, rubber, or other nonmetallics are part of a component or assembly, caution shall be exercised in baking the dry-film coating. Infra-red lamps placed in a judicious manner are preferred to an oven for these vulnerable materials.

NOTICE: When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodians

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

PREPARATION AND APPLICATION OF MLF-5, DRY FILM LUBRICANT

1. SCOPE

This procedure describes the requirements for preparation and application of MLF-5, a dry film lubricant, that is LOX compatible, and has a low coefficient of friction which is capable of supporting high loads without penetration.

2. This procedure conforms to the following specifications:

Federal

RR-S-366	Sieves, Standard for testing purposes
Fed-Std-209A	Federal Clean Room and Work Station Requirements and Controlled Environment

Military

MIL-M-7866	Molybdenum Disulfide, Technical Lubrication Grade
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George C. Marshall Space Flight Center

MSFC-PROC-404	Gases, Drying and Preservation
MSFC-SPEC-164	Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic Systems

3. REQUIREMENTS

3.1 Facility — The environment of the facility must be compatible with the cleanliness levels of the end items.

3.1.1 Facility requirement — Class 30 000 and Class 100 000 Clean Rooms

3.1.2 Exhaust Hood

3.1.3 Laminar Flow Bench

- 3.1.4 Lighting — as required by Fed-Std-209A
- 3.1.5 Airlock Entrance
- 3.1.6 Missile Air Supply
- 3.2 Chemical
 - 3.2.1 Molybdenum disulfide size less than No. 44 micron
 - 3.2.2 Graphite powder — size less than No. 44 micron
 - 3.2.3 Gold dust, pure, size — less than No. 44 micron
 - 3.2.4 Sodium Silicate solution, Na_2O ; SiO_2 , at ratio 1.2 to 0.9 — 47° Baume
 - 3.2.5 NPX, Turgitol, non-ionic
 - 3.2.6 Water — Distilled pH 6.0 to 7.5; solids 0.2 mg/100cc
- 3.3 Equipment
 - 3.3.1 Beakers
 - 3.3.2 Rinse Bottles
 - 3.3.3 Magnetic Stirrer
 - 3.3.4 Spray Equipment
 - 3.3.5 Microscope
 - 3.3.6 Tweezers
 - 3.3.7 Motorized Equipment for rotating parts
 - 3.3.8 Variac (variable resistor)
 - 3.3.9 Oven
 - 3.3.10 Micrometer

3.3.11 Racks or holding equipment

3.3.12 Nylon gloves or equivalent

3.3.13 Nylon wrapping material

3.3.14 High speed blender

3.3.15 Heat lamps

3.3.16 Falex test specimens

3.3.17 Maskant tape (Government Stock No. 7510-551-9823 or equal)

3.3.18 Dry grit blast equipment

3.3.19 Overspray protective shielding

3.3.20 Desiccator cabinet

3.4 Qualification:

3.4.1 Qualification of Facility — The qualification of facility should comply with paragraph 3.1.

3.4.2 Qualification of Process — The process will be qualified by this Manufacturing Procedure: To include qualification of procured new lots.

3.4.3 Qualification of Operator — Each spray operator will be qualified by demonstrating his skill before spraying production parts.

3.4.4 Requirements for qualifying facility, process, lubricant operator, are as follows:

3.4.4.1 Facility and Process — 20 Falex life endurance test samples

3.4.4.2 Operator — 3 Falex life endurance test samples

3.4.4.3 Lubricant — 3 Falex life endurance test samples (New raw material lots)

3.4.4.4 Sampling (once a month) — 3 Falex life endurance test samples

3.4.5 Qualification of MLF-5 is performed jointly by the following sections:

S&E-ME-DPP — Responsible for mixing MLF-5 lubricant.

S&E-ME-DFP — Responsible for application of MLF-5 lubricant.

S&E-QUAL-AAS — Responsible for qualifying MLF-5 lubricant.

3.4.6 Qualification requirements are:

3.4.6.1 Endurance Life Test — The bonded lubricant shall have an average endurance life of:

3.4.6.2 Class I — Heavy Load — greater than 8 minutes.

Class II — Light Load — greater than 4 minutes.

3.4.6.3 Pot Life — The maximum time after mixing of MLF-5 is 5 days. No single sample shall fail in less than 3 minutes.

3.5 Packaging — Parts shall be double bag sealed with inner bag being Nylon 6 and outer bag being Polyethylene (no further cleaning required after packing).

3.6 Surface Finish — The surface finish of areas to be lubricated, before and after grit blasting, shall be specified on detail drawings. It is recommended that surface finish be machined to a roughness of 13 or less microinches (rms), and a surface finish of 16 to 30 microinches (rms) after grit blasting. Care should be exercised to minimize metal removal while grit blasting.

3.7 Cleanliness — Shall be specified by MSFC-SPEC-164.

NOTE: (1) Generally, 0.0002 to 0.0005 inch of surface may be removed.

(2) All parts should be lubricated within 24 hours after cleaning.

4. Mixture of MLF-5 Lubricant

4.1 Contents and proportions are:

<u>Ingredients</u>	<u>Parts by Weight</u>
Molybdenum disulfide	10
Graphite	1
Gold	5
Sodium silicate solution	7
Distilled water	60 (see note)
NPX, Turgitol	1 drop per 60 ml of distilled water

NOTE: Additional quantities of distilled water up to 30 parts by weight, may be added, as required, to maintain proper flow of MLF-5 through spray nozzle.

4.2 Procedure for Mixing MLF-5 Lubricant

4.2.1 MLF-5 lubricant is mixed as follows:

4.2.1.1 Add 10 grams of molybdenum disulfide into a 250-cc beaker

4.2.1.2 Add 1 gram of graphite

4.2.1.3 Add 7 grams of sodium silicate solution

4.2.1.4 Add 20 cc of distilled water and mix thoroughly with magnetic stirrer.

4.2.1.5 Transfer content of beaker into a blender cup, then rinse beaker with about 15 cc distilled water.

NOTE: Larger amounts of Lubricant may be mixed as required.

4.2.1.6 The mixture should be mixed with a blender for 5 minutes, with a mixing cycle of 1 minute mix to 1 minute cool, for a total of 5 cycles.

4.2.1.7 Transfer mixture from blender cup to spray flask. Rinse beaker cup with 3 rinsings of 10 cc of distilled water, then place mixture on magnetic stirrer.

4.2.1.8 With magnetic stirrer on medium speed, slowly add 5 grams of gold dust to mixture. Mix for 10 minutes. Lubricant is ready for spray application.

4.2.1.9 The prepared lubricant should be stored at ambient temperature, and should not be used after 5 days have expired from time of mix.

5. Cleaning Process

5.1 Precleaning — Remove oil and/or grit with cloth moistened with acetone or MEK.

5.2 Degrease — Vapor degrease with trichloroethylene.

5.3 Surface finish — Surface finish with grit blast per paragraph 3.5.

5.4 Final cleaning

5.4.1 Stainless Steel — Per MS-100.9A, Cleaning of Miscellaneous Components (see note).

5.4.2 Aluminum — Per MS-100.9A, Cleaning of Miscellaneous Components (see note)

5.4.3 Test Specimens — Degrease and ultrasonically clean in Freon (see note)

NOTE: (1) Packaging — Substitute Nylon "6" for aluminum foil

(2) After cleaning, parts are placed in Desiccator Cabinet until required for measurements and/or MLF-5 application.

5.5 Preserving of clean parts — Preservation of parts is covered under packaging per paragraph 5.4.

6. Application of Lubricant

6.1 Environment must be compatible with cleanliness of final production parts.

6.2 Pre-heating — Production parts should be preheated to a constant temperature of 150° F before lubricant is applied.

6.3 Application

6.3.1 The MLF-5 shall be applied to the designated surface of the part as a fine mist, thus preventing moist spots. The thickness of each coating should be 0.0001 to 0.0004 inch. Parts should be shielded from overspray.

6.3.2 Each coating should be dried before additional coats are applied. An oven or infrared lamp is sufficient for drying of parts between spray operation; however, the temperature should not exceed 150°F.

6.3.3 Thickness — The thickness of the MLF-5 shall be specified by a drawing; however, the recommended thickness of production parts and/or specimens should be 0.0004 to 0.0012 inch.

6.3.4 The optimum distance of spray nozzle from part is 20 to 22 inches.

6.3.5 Curing Process — The curing steps are:

<u>STEP</u>	<u>TEMPERATURE</u>	<u>TIME</u>
1	82°C (180°F)	2 hours
2	149°C (300°F)	8 hours
3	Reduce slowly from 149°C to ambient temperature	

6.4 Workmanship — The coating of MLF-5 should be smooth and uniform in color. Any cracks, scratches, pin holes, etc., would constitute rejection of parts. Questionable areas are to be examined with 12X magnification.

6.5 Packaging — Lubricated parts shall be double bag sealed after the lubricated parts have remained in desiccator for 24 hours, with the inner bag being Nylon 6 and outer bag being polyethylene.

6.6 Endurance Test — See paragraph 3.4.6.1. Test specimen shall be by S&E-QUAL-AAS's Falex Procedure. Every sample must pass the 4-minute test.

7. Quality Assurance

7.1 Qualification of production items.

7.1.1 Falex test specimen shall accompany production parts to S&E-QUAL-AAS.

7.1.2 The Falex test specimen will be examined for workmanship, film thickness, adhesion, and endurance.

7.2 Certification — S&E-QUAL-AAS will notify S&E-ME-DFP of the certification of production parts.

MANUFACTURING SPECIFICATION FOR
THE DRYING OF COMPONENTS IN A VACUUM

1. SCOPE

1.1 Scope - This manufacturing specification covers the approved PE Laboratory requirements for vacuum drying metallic and nonmetallic components for use in space launch vehicle LOX or fuel applications.

1.2 Classification - The drying methods covered by this specification shall be of the following classes:

1.2.1 Class I 180° F (±20°) and 5 to 50 ml of mercury vacuum - Class I vacuum drying is to be employed for ceramic components, for bare uncoated metallic components, Teflon or Kel-F components, and for components which are combinations of ceramics or metals with Teflon or Kel-F. Examples of such components are:

- Gimbal blocks
- Aluminum or stainless steel fittings
- Nuts, bolts, clamps

1.2.2 Class II 120° F (±10°) and 5 to 25 ml of mercury vacuum - Class II vacuum drying is to be employed for all space launch vehicle components fabricated from combinations of metals and nonmetals and which require drying after cold tests. Examples of such components are:

- LOX relief valve
- LOX fill and drain valve
- LOX replenishing valve

1. 2. 3 Class III [110° F (±10°) and 5 to 25 mm of Hg] - Class III vacuum drying is to be employed for nonmetallic components and for metallic components combined with nonmetallics other than Teflon or Kel-F. Examples of such components are:

- Accumulator bladder
- All types of O-rings
- All rubber materials, synthetic or natural

2. APPLICABLE DOCUMENTS

2. 1 Governmental - The following documents, of the issue in effect on the date of use of this specification, form a part of this specification to the extent indicated herein.

SPECIFICATIONS

Federal

BB-N-411a	Nitrogen
UU-T-81d(1)	Tags, Shipping and Stock
PPP-T-60(2)	Tape; Pressure Sensitive Adhesive, Waterproof

Military

MIL-A-00148b(ORD)	Aluminum Foil
MIL-H-19457(3)	Hydraulic Fluid, Fire Resistant

George C. Marshall Space Flight Center

10509305	Cleanliness of Components for Use in Liquid Oxygen, Fuel and Pneumatic Systems
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DRAWINGS

George C. Marshall Space Flight Center

10414000	Tool, Testing, for LOX Relief Valve
10414002	Fixture, Pressure Test, LOX Drain and Fill Valve
10414003	Fixture, Test, LOX Replenishing Valve Assembly

2.2 Other Publications - The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

Celanese Corporation of America

Product Bulletin S-49-1	Cellulube Safety Series Fire-Resistant Hydraulic Fluids and Synthetic Lubricants in Controlled Viscosities (Second Edition)
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(Copies of the publication listed above may be obtained from the Celanese Chemical Company, 3130 Maple Drive N.E., Atlanta 5, Georgia.)

3. REQUIREMENTS

3.1 General - No deviation from the requirements of this manufacturing specification shall be submitted without prior written approval of the MSFC initiating activity.

3.2 Equipment

3.2.1 Vacuum chamber - The vacuum chamber used shall be capable of being evacuated within 110 (± 10) minutes to a vacuum of 5 to 25 mm of mercury and shall not show a vacuum loss of more than 5 mm of mercury when tested by blanking off from its vacuum pump for a period of 30 (± 5) minutes. It shall be equipped with suitable infra-red heating elements capable of heating a 2000-pound work load from 70 to 250° F ($\pm 10^\circ$) within 30 minutes.

3. 2. 2 Vacuum pump - The vacuum pump used shall be a synthetic lubricant sealed, rotary-piston type, mechanical vacuum pump, (Kinney KDH-530 or equal), capable of lowering the pressure in the vacuum chamber, containing a 2000-pound work load of miscellaneous components, from atmospheric pressure to within the range of 5 to 25 millimeters of mercury, with a work load temperature of 250° F ($\pm 10^\circ$), in an evacuation time of 2 hours maximum.

3. 3 Materials

3. 3. 1 Drying or preservation gas - Gases used in pre-drying or preservation processes shall be filtered to a 100-micron level (absolute), the oil content shall be not greater than 3 parts per million by weight, and the moisture content shall not be greater than 24 parts per million.

3. 3. 2 Aluminum foil - Aluminum foil used to protect cleaned, dried surfaces shall be of the dry annealed type conforming to paragraph 6. 3. 2 of Specification MIL-A-00148b.

3. 3. 3 Tape - Tape used to secure and reinforce protective coverings shall be Type I, Class 1 or 3 of Specification PPP-T-60.

3. 3. 4 Tags - Components dried by a vacuum method shall be identified by tags conforming to Specification UU-T-81d(1), Type C.

3. 3. 5 Vacuum pump lubricant and sealant - The vacuum pump lubricant-sealant used shall be a synthetic type compatible with liquid oxygen (Cellulube 550, or equal) and shall meet all the applicable requirements of Specification MIL-H-19457(3) with the exception of pour point, viscosity, and color.

3. 4 Predrying - All flexible tubing, and all components having internal or external configurations which might hamper the extraction of vaporized cleaning liquids from their surfaces, shall be predried using air or a compressed gas. The air or gas used shall be preheated to the same maximum temperature to be used in the final drying operation; 180° F ($\pm 20^\circ$) for Class I, 120° F ($\pm 10^\circ$) for Class II, and 110° F ($\pm 10^\circ$) for Class III; as determined by the material from which the component is constructed (see paragraph 1. 2). Predrying shall be continued for a minimum of 30 minutes and a maximum of 60 minutes.

3. 5 Final Drying Procedures

3.5.1 Class I drying

3.5.1.1 Inspection - Inspect each component to assure that it has been pre-dried if necessary (see paragraph 3.4) and to determine that the materials used in its construction are materials which will not be damaged by exposure to Class I drying temperatures. (see paragraph 1.2).

3.5.1.2 Loading into chamber - Place components in the vacuum chamber and position the temperature probe at the center of the load to insure attainment of the desired temperature. Close and vacuum-seal the opening through which the components were placed in the chamber.

3.5.1.3 Preliminary evacuation - To expedite the rapid heating of the work load by leaving air present for convection, partially evacuate the vacuum chamber to approximately 740 mm of mercury absolute.

3.5.1.4 Heating - Set the vacuum chamber temperature controls to 180°F, turn on power to heating elements and raise the temperature of the work load in the chamber to 180°F ($\pm 20^\circ$).

3.5.1.5 Final evacuation - As quickly as the work load attains a temperature of 180°F ($\pm 20^\circ$), evacuate the chamber to a vacuum within the range of 5 to 50 mm of mercury absolute.

3.5.1.6 Drying - Allow predried components to remain at a temperature of 180°F ($\pm 20^\circ$) and at a vacuum of 5 to 50 mm of mercury for a minimum of 30 minutes. Allow components which were not predried to remain at a temperature of 180°F ($\pm 20^\circ$) and at a vacuum of 5 to 50 mm of mercury for a minimum of 60 minutes.

3.5.1.7 Removal from chamber - At the completion of the drying cycle, shut off the vacuum pump and turn off the power to the heating elements. Reestablish atmospheric pressure inside the vacuum chamber by slowly admitting air which meets the requirements of paragraph 3.3.1. Open vacuum chamber and remove dried components.

3.5.2 Class II drying

3.5.2.1 Inspection - Inspect each component to assure that it has been pre-dried if necessary (see paragraph 3.4) and to determine that the materials

used in its construction are materials which will not be damaged by exposure to Class II drying temperatures (see paragraph 1. 2).

3. 5. 2. 2 Loading into chamber - Load components into vacuum in the manner specified in paragraph 3. 5. 1. 2.

3. 5. 2. 3 Preliminary evacuation - Evacuate the vacuum chamber in the manner specified in paragraph 3. 5. 1. 3.

3. 5. 2. 4 Heating - Set the vacuum chamber temperature controls to 120° F, turn on power to heating elements and raise the temperature of the work load in the chamber to 120° F ($\pm 10^\circ$).

3. 5. 2. 5 Final evacuation - As quickly as the work load attains a temperature of 120° F ($\pm 10^\circ$), evacuate the chamber to a vacuum within the range of 5 to 25 mm of mercury.

3. 5. 2. 6 Drying - Allow predried components to remain at a temperature of 120° F ($\pm 10^\circ$) and at a vacuum of 5 to 25 mm of mercury for a minimum of 30 minutes. Allow components which were not predried to remain at a temperature of 120° F ($\pm 10^\circ$) and at a vacuum of 5 to 25 mm of mercury for a minimum of 60 minutes.

3. 5. 2. 7 Removal from chamber - Remove components from the vacuum chamber in the manner specified in paragraph 3. 5. 1. 7.

3. 5. 3 Class III drying

3. 5. 3. 1 Inspection - Inspect each component to assure that it has been predried if necessary (see paragraph 3. 4) and to determine that the materials used in its construction are materials which will not be damaged by exposure to Class III drying temperatures (see paragraph 1. 2).

3. 5. 3. 2 Loading into chamber - Load components into vacuum chamber in the manner specified in paragraph 3. 5. 1. 2.

3. 5. 3. 3 Preliminary evacuation - Evacuate the vacuum chamber in the manner specified in paragraph 3. 5. 1. 3.

3.5.3.4 Heating - Set the vacuum chamber temperature controls to 110° F, turn on power to heating elements, and raise the temperature of the work load in the chamber to 110° F ($\pm 10^\circ$).

3.5.3.5 Final evacuation - As quickly as the work load attains a temperature of 110° F ($\pm 10^\circ$), and evacuate the chamber to a vacuum within the range of 5 to 25 mm of mercury.

3.5.3.6 Drying - Allow predried components to remain at a temperature of 100° F ($\pm 10^\circ$) and at a vacuum of 5 to 25 mm mercury for a minimum of 30 minutes. Allow components which were not predried to remain at a temperature of 110° F ($\pm 10^\circ$) and at a vacuum of 5 to 25 mm of mercury for a minimum of 60 minutes.

3.5.3.7 Removal from chamber - Remove components from the vacuum chamber in the manner specified in paragraph 3.5.1.7.

3.6 Cleanliness - The final cleanliness level of components vacuum dried according to the provisions of this specification is dependent upon the cleanliness level of the vacuum chamber and the particle size of the pruging gas. The provisions of paragraphs 3.3 through 3.5 adequately meet the drying requirements for LOX and fuel systems. If more stringent requirements become necessary, a more restrictive procedure will be required.

4. MANUFACTURING CONTROL PROVISIONS

4.1 Equipment - Care shall be exercised to detect any malfunctioning of vacuum system equipment during daily operation. At least once each week, temperature sensing devices and vacuum gages shall be checked under operating conditions and their accuracies verified by comparison with the readings obtained from calibrated test temperature and vacuum sensing elements. The test temperature-sensing and the vacuum-sensing elements shall have been calibrated against Bureau of Standards primary or secondary certified elements within the immediately preceding three month period.

4.2 Dryness - Component dryness shall be determined by passing drying gas (see paragraph 3.3.1) through or over the component surfaces and monitoring the dew point of the gas before and after contacting the component. No increase in moisture content of the drying gas shall be allowed.

4.3 Cleanliness - The cleanliness of vacuum-dried components shall be determined for particle size and nonvolatile residue in the manner specified in paragraphs 4.4.2.1 and 4.4.2.2, respectively, of Specification 10509305.

5. PREPARATION FOR DELIVERY

5.1 Preservation and Packaging - Vacuum-dried components shall be protected from recontamination by covering their surfaces or openings with a minimum of two layers of aluminum foil secured and reinforced with tape. The protected components shall then be placed in LOX-clean polyethylene bags, the bags purged with drying gas, then heat-sealed to assure an inert package during storage. When secured closures are used in openings instead of aluminum foil coverings, the closure material shall be compatible with material of the component in which it is used.

5.2 Marking - Tags attached to each vacuum dried component shall contain the following information:

- Part or identification number
- Contractor identification
- Method of drying and particle size micron level
- Date of drying
- Service medium or intended use of component
- Manufacturer's serial number
- Any additional information required by contract

6. NOTES

6.1 Intended Use - This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use in the vacuum drying of metallic and nonmetallic components for use in space launch vehicle applications.

NOTICE; When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government

procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

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SECTION V. STAINLESS ALLOYS

The chemical processes in this section were formulated and used successfully on the Saturn Program. All existing documents related to chemical processing of stainless steels are contained in this section. Copies of specifications, standards, drawings, and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.

5.1 CLEANING SOLVENTS

Cleaning of Unlined Stainless Steel Bellows - MS 100.5A

Carbon Removal - MS 100.9A

Carbon Removal from Heat Exchangers - MPD 25003

5.2 CLEANING OF TUBING AND FLEXIBLE HOSE

Cleaning of Stainless Steel Tubing Ends - MPD 25009

Cleaning Tubing for Brazing - MPD 26301C

Cleaning Tubing Attached to Instruments - MPD 26302

Cleaning of Unlined Bellows - MS 100.5A

Specifications for Protection of Pipe and Tubing During Welding - MS 200.2A

5.3 CLEANING, PICKLING, AND PASSIVATING

Cleaning and Passivation of Heat Exchangers - MS 100.3C

Cleaning and Passivation of 300 Series Stainless Steel - MS 100.4A

5.4 ELECTROLYTIC CLEANING AND POLISHING

Electropolishing of Corrosion Resistant Steel - MS 150.4

5.5 CLEANING MISCELLANEOUS MATERIAL/COMPONENTS

Cleaning of Control Assemblies - MS 100.7A

Cleaning and Testing of Components in Fuel Systems -
M-ME-MPROC 103.0

Cleaning Electrical Connectors - M-ME-MPROC 104.0

Cleaning and Testing of Hydraulic Systems - M-ME-MPROC 105.0

Cleaning LOX Suction Lines - MPD 25007

Cleaning and Polishing Optical Experiment Chamber - MPD 25018

Cleaning of Filter Elements - MPD 25019

Cleaning ATM Black Boxes - MPD 26003A

Cleaning Cryogenic Test Tank - MPD 26614

Cleaning Temperature Transducer - MPD 26632

Cleaning ATM Opto-Mechanical Assemblies - MPD 26634

MANUFACTURING SPECIFICATIONS FOR CLEANING
OF UNLINED STAINLESS STEEL BELLOWS

1. SCOPE

1.1 Scope

This specification covers the approved PE Laboratory manufacturing requirements for attaining the cleanliness standards for unlined stainless steel bellows used on space launch vehicles.

1.2 Applicability

1.2.1 Stainless steel grades

Cleaning requirements set forth in this specification shall be applicable to bellows made of the following grades of stainless steel; 304, 316, 321, and 347.

1.2.2 Types

This specification may apply to stainless steel bellows joined to aluminum flanges, as well as to those consisting only of stainless steel, provided the aluminum is protected during processing. This specification does not apply to bellows which are not sealed by cap welding, or any bellows that cannot be positively dried by vacuum-oven drying.

2. APPLICABLE DOCUMENTS

2.1 Governmental

The following documents, of the issue in effect on the date of use of this specification, form a part of this specification.

SPECIFICATIONS

Federal

O-A-88 (1)

Acid, Nitric, Technical Grade

O-H-795	Acid Hydrofluoric, Technical Grade
O-P-94a	Paper, Test; pH Indicator
O-T-634a	Trichloroethylene, Technical
BB-N-411a	Nitrogen
UU-T-81d	Tags, Shipping and Stock
PPP-T-69 (2)	Tape, Pressure Sensitive Adhesive, Waterproof for Packaging and Sealing

Military

MIL-A-00148 (ORD)	Aluminum Foil, Dry Annealed
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George C. Marshall Space Flight Center

MSFC-SPEC-164	Cleanliness of Components for use in Liquid Oxygen Fuel and Pneumatic Systems
MS 150.0	Manufacturing Specification for the Vapor Degreasing of Metallic Surfaces

2.2 Other Publications

The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

TURCO PRODUCTS, INC.

Tech. Product Data Bulletin No. 76	Turco Vitro-Klene
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(Copies of the publication listed above may be obtained from Turco Products, Inc., 6135 S. Central Avenue, Los Angeles 1, California.)

FIDELITY CHEMICAL PRODUCTS CORP.

Plastic Coating Compound
Strippable Hot Dip Type

(Copies of the publication listed above may be obtained from the Fidelity Chemical Products Corporation, Newark, New Jersey.)

3. REQUIREMENTS

3.1 General

No deviation from the requirements of this manufacturing specification shall be permitted without the prior written approval of the MSFC initiating activity.

3.2 Materials

3.2.1 Vapor degreasing solvent

Trichloroethylene used in this process shall meet the requirements of Type II of Specification O-T-634a.

3.2.2 Test solvent

Trichloroethylene used for testing the cleanliness of LOX components shall meet the requirements of Type I of Specification O-T-634a, except that the nonvolatile residue shall not be greater than 0.010 gm. per 500 ml when the solution is heated to 221 to 230° F and that no particle larger than 175 microns in any dimension shall be present.

3.2.3 Demineralized water

Demineralized water shall be filtered to remove all particles larger than 175 microns and shall possess a minimum electrical resistivity of 50 000 ohms.

3.2.4 Alkaline cleaner

The alkaline cleaner used for degreasing of metals in this process shall be equal in quality of cleaning to Turco Vitro-Klene.

3.2.5 Cellulose acetate

The cellulose acetate employed for protecting metals or areas from attack by the chemicals employed in this process shall be equal in quality to Fidelity Chemical Products Corporation, Plastic Coating Compound.

3.2.6 Aluminum foil

Dry-annealed aluminum foil employed in this process for protecting cleaned bellows against contamination shall conform to paragraph 6.3.2 of Specification MIL-A-00148 (ORD).

3.2.7 Tags

Tags employed in marking of cleaned components shall conform to style C of Specification UU- T-81d.

3.2.8 pH indicator

The pH indicating paper used in this process for checking neutrality shall be of the Medium range, Class A. of Specification O-P-94a.

3.2.9 Drying and preservation gases

The nitrogen or air employed for drying or preservation shall be filtered to a 100 micron level, possess an oil content not greater than 3 parts per million (by weight), and contain a maximum of 24 parts per million (by volume) of moisture.

3.3 Drying

3.3.1 Oven drying

When drying is performed in an oven, the operating temperature shall be 180° F ($\pm 20^\circ$) and the oven shall be purged with gas meeting the requirements of paragraph 3.2.9 of this specification.

3.3.2 Vacuum-oven drying

The operating temperature for vacuum-oven drying shall be 180° F ($\pm 20^\circ$). The vacuum, as read on the alphanon gauge, shall be 740 mm of mercury and shall exist for 1 hour at temperature. Atmospheric pressure shall be restored by admitting air meeting paragraph 3.2.9 of this specification.

3.3.3 Compressed, heated gases

When drying is performed by blasting or purging a component with heated, compressed gases, the pressure shall be 60 (± 20) psi and the temperature shall be 180° F ($\pm 20^\circ$). The gas shall conform to paragraph 3.2.9 of this specification.

3.4 Flanged stainless steel bellows

This procedure covers unlined bellows or subassemblies made only of stainless steel.

3.4.1 Inspection

Visually inspect each part to determine its cleanliness and record any unusual contamination.

3.4.2 Vapor degrease

Vapor degrease per MS 150.0.

3.4.3 Hot alkaline clean

Immerse in an aqueous solution of Turco Vitro-Klene heated to 200° F ($\pm 5^\circ$) for 10 to 15 minutes. Vitro-Klene concentration in solution shall be 8 to 10 ounces per gallon of water.

3.4.4 Hot water rinse

Rinse in hot water at 180° F ($\pm 10^\circ$).

3.4.5 Spray

Spray rinse in cold water for 3 to 5 minutes.

3.4.6 Pickle

Immerse each unit for 20 to 30 minutes in an acid solution of 20 (± 5) percent acid (by weight) operating at 150° F ($\pm 10^\circ$).

3.4.7 Rinse

Rinse in tank with overflowing water for 10 to 15 minutes at ambient temperature.

3.4.8 Passivate

Immerse each unit for 15 to 20 minutes in a 20 (± 5) percent nitric acid (by volume) and 2 (± 0.5) percent potassium dichromate (by weight) solution at 125° F ($\pm 5^\circ$).

3.4.9 Rinse

Rinse in demineralized water for 15 to 20 minutes at ambient temperature.

3.4.10 Spray

Spray rinse with demineralized water for 3 to 5 minutes at ambient temperature until a pH of 6 to 8 is reached.

3.4.11 Dry

Dry with pressurized hot gas per paragraph 3.3.3 of this specification.

3.4.12 Oven

Oven dry per paragraph 3.3.1 of this specification.

3.4.13 Degrease

Rinse each unit for at least 5 minutes with trichloroethylene suitable as a test liquid which is covered in paragraph 3.2.4 of this specification. Repeat this rinse for two more rinsings. Sample 500 cubic centimeters of effluent into LOX clean containers and analyze per paragraph 4.4.2 of Specification MSFC-SPEC-164 for nonvolatile residue and particle size.

NOTE: Perform this operation under a fume exhaust hood. Use gloves and respirator if a fume exhaust hood is not available.

3.4.14 Dry

Dry with pressurized hot gas per paragraph 3.3.3 of this specification.

3.4.15 Vacuum dry

Vacuum dry per paragraph 3.3.2 of this specification.

3.4.16 Inspect

Check bellows visually for cleanliness and thorough dryness.

3.4.17 Preserve and package

Bellows should be protected and packaged per Specification MSFC-SPEC-164.

3.4.2 Aluminum flanged stainless steel bellows

This procedure applies to unlined stainless steel bellows with aluminum flanges.

3.4.2.1 Vapor degrease

Vapor degrease per Specification MS 150.0.

3.4.2.2 Quick-dip

Coat each aluminum flange or area desired to be protected with hot plastic coating compound by dip immersion at 350° F ($\pm 10^\circ$). Allow to cool and harden.

3.4.2.3 Hot alkaline clean

Immerse in an aqueous solution of Turco Vitro-Klene heated to 200° F ($\pm 5^\circ$) for 10 to 15 minutes. Vitro-Klene concentration in the solution should be from 8 to 10 ounces per gallon of water.

3.4.2.4 Hot water rinse

Rinse in hot water at 180° F ($\pm 10^\circ$).

3.4.2.5 Spray

Spray rinse in cold water for 3 to 5 minutes.

3.4.2.6 Pickle

Immerse each unit for 20 to 30 minutes in an acid solution of 20 (± 5) percent nitric acid (by weight) and 1.5 (± 0.5) percent hydrofluoric acid (by weight) operating at 150° F ($\pm 10^\circ$).

3.4.2.7 Rinse

Rinse in a tank with overflowing water for 10 to 15 minutes at ambient temperature.

3.4.2.8 Passivate

Immerse each unit for 15 to 20 minutes in a solution of 20 (\pm 5) percent nitric acid (by weight) and 2 (\pm 0.5) percent potassium dichromate (by weight) at 120°F (\pm 5°).

3.4.2.9 Rinse

Rinse in demineralized water for 15 to 20 minutes at ambient temperature.

3.4.2.10 Spray

Spray rinse with demineralized water for 3 to 5 minutes at ambient temperature until a pH of 6 to 8 is attained.

3.4.2.11 Dry

Dry with pressurized hot gas per paragraph 3.3.3 of this specification.

3.4.2.12 Stripping

Completely strip the plastic coating compound off the flanges or other protected areas.

3.4.2.13 Rinse

Rinse the unit in demineralized water for 2 to 5 minutes at ambient temperature.

3.4.2.14 Dry

Dry with pressurized hot gas per paragraph 3.3.3 of this specification.

3.4.2.15 Oven

Vacuum dry per paragraph 3.3.1 of this specification.

3.4.2.16 Degrease

Rinse each unit for at least 5 minutes with Trichloroethylene suitable as a test solvent and which is covered in paragraph 3.2.4 of this specification.

Repeat this rinsing for two more rinsings. Sample 500 cubic centimeters of the effluent into LOX clean containers and analyze per paragraph 4.4.2 of Specification MSFC-SPEC-164 for nonvolatile residue and particle size. (See note paragraph 3.4.1.13).

3.4.2.17 Dry

Dry with pressurized hot gas per paragraph 3.3.3 of this specification.

3.4.2.18 Vacuum dry

Vacuum dry per paragraph 3.3.2 of this specification.

3.4.2.19 Inspect

Check bellows visually for cleanliness and thorough dryness.

3.4.2.20 Preserve and package

Bellows should be protected and packaged per Specification MSFC-SPEC-164.

3.4.3 Recleaning

If the additional cleaning or recleaning of unlined stainless steel bellows becomes necessary, they must be cleaned to the original cleanliness level by following the procedure outlined below.

3.4.3.1 Operation

Follow paragraphs 3.4.2.16 through 3.4.2.20.

4. QUALITY ASSURANCE PROVISIONS

4.1 Level of Cleanliness

Components cleaned by this procedure shall meet the requirements of Specification MSFC-SPEC-164.

4.2 Solution Maintenance

Tank solutions employed in alkaline degreasing, pickling, and passivating shall be analyzed periodically to preserve efficacy by maintaining their compositions within operating limits.

4.3 Quality Control

Control of the quality of various tank operations can be assured by adherence to the time and temperature specified.

5. PREPARATION FOR DELIVERY

5.1 Preservation and Packing

The sealed, finished bellows shall be packaged and preserved in accordance with Specification MSFC-SPEC-164, paragraph 3.6.1.5.

5.2 Marking

When applicable, the tags attached to the sealed bellows shall record the following information:

- Part or identification number
- Contractor identification
- Method of cleaning & particle size, micron level
- Date of cleaning
- Service medium or intended use of component
- Manufacturer's serial number
- Any further information required by contract

6. NOTES

6.1 Intended Use

This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use in cleaning of unlined stainless steel bellows for space launch vehicle applications.

NOTICE: When government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related government procurement operation, the United States Government incurs no responsibility

nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

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MANUFACTURING SPECIFICATIONS FOR
CLEANING OF MISCELLANEOUS COMPONENTS

1. SCOPE

1.1 Scope

This specification covers the approved PE Laboratory manufacturing requirements for the cleaning of miscellaneous metallic components and combinations which are not specifically covered by another manufacturing specification.

1.2 Applicability

Cleaning of miscellaneous components shall be accomplished by the four processes listed below. Each process is designed to encompass a large number of components.

1.2.1 Process 1

This process is to be employed for bare (uncoated) metallic components, Teflon or Kel-F components, and for components which are combinations of metals with Teflon or Kel-F. Examples of items to be cleaned by this process are:

- Aluminum or stainless steel fittings
- Nuts, bolts, clamps, etc.

NOTE: Anodized aluminum fittings are an exception of Process 1.

1.2.2 Process 2

This process is to be employed for metallic components having anodized, parkerized, or plated surfaces which shall not be subjected to ultrasonic cleaning. Examples of items to be cleaned by this process are:

- Injector plate
- LOX dome

- Gimbal blocks
- All anodized, parkerized, or any other type of finish which could be detrimentally affected by the use of ultrasonic cleaning.

1.2.3 Process 3

This process is to be employed for nonmetallic components or metallic components combined with nonmetallics other than Teflon or Kel-F, which shall not be subjected to ultrasonics. Examples of these items are:

- Accumulator bladder
- All types of O-rings.
- All rubber material, synthetic or natural
- Molded rubber seal with anodized aluminum
- Plenum chamber

1.2.4 Process 4

This process is to be employed for specially designed flight hardware such as a continuous level probe and any other hardware, applicable to the following procedure. (Refer to paragraph 3.3.4)

2. APPLICABLE DOCUMENTS

2.1 Governmental

The following documents, of the issue in effect on the date of use of this specification, form a part of this specification.

Federal

O-A-51c	Acetone, Technical
O-E-760b	Ethyl Alcohol (Ethanol), Denatured Alcohol, and Proprietary Solvent
O-P-313	Phosphoric Acid; Technical Grade

O-T-634a	Trichloroethylene, Technical
BB-N-411a	Nitrogen
UU-T-81F(1)	Tags, Shipping and Stock
PPP-T-60(3)	Tape, Pressure Sensitive Adhesive, Waterproof

Military

MIL-A-00148C(2)	Aluminum Foil
MIL-P-3803	Plastic, Polyethylene, Molded and Extruded Shapes, Sheet and Tubing
MIL-D-16791C	Detergent, Nonionic

George C. Marshall Space Flight Center

MSFC-SPEC-164	Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic Systems, Specification for
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3. REQUIREMENTS

3.1 General

No deviation from the requirements of this manufacturing specification shall be permitted without the prior written approval of the MSFC initiating activity.

3.2 Materials

3.2.1 Vapor cleaning solvent

Trichloroethylene use for vapor degreasing shall meet the requirements of Type II of Specification O-T-634a.

3.2.2 Liquid cleaning solvent

The liquid trichloroethylene used for immersion or flushing shall meet the requirements of Type I of Specification O-T-634a, with the further require-

ment that the nonvolatile residue shall be not greater than 0.010 gram per 500 milliliters when evaporated as specified in Specification MSFC-SPEC-164. There shall be no particle over 175 microns in any dimension and no more than 5 particles from 100 to 175 microns in size per 500 ml of test solvent.

3.2.3 Drying or preservation gases

Nitrogen or air used for the drying or preservation of components shall contain no particle larger than 100 microns in any dimension. The oil content shall be not greater than 3 parts per million (by weight), and the moisture content shall not be greater than 24 parts per million.

3.2.4 Demineralized water

Demineralized water shall contain no particles larger than 175 microns per 500 ml. It shall also have a minimum electrical resistivity of 50,000 ohms. pH of water shall be between 6 and 8.

3.2.5 Aluminum foil

Dry annealed aluminum foil used to protect cleaned, dried surfaces shall conform to paragraph 6.3.2 of Specification MIL-A-00148C(2).

3.2.6 Tape

Tape used to secure protective coverings shall conform to Specification PPP-T-60(3).

3.2.7 Nonionic detergent

The detergent used in these processes shall be nonionic and meet the requirements of Specification MIL-D-16791C.

3.2.8 Tags

Components shall be identified by tags conforming to Specification UU-T-81F(1), Type C, when required.

3.2.9 Phosphoric Acid

Phosphoric acid used in cleaning components shall conform to Grade A of Specification O-P-313.

3.2.10 Alcohol

The alcohol used in cleaning shall conform to Specification O-E-760B.

3.2.11 Acetone

Acetone used in cleaning shall conform to Specification O-A-51c.

3.3 Procedure

3.3.1 Process 1

Uncoated metallic parts and Teflon, or Kel-F parts shall be cleaned by the following process:

3.3.1.1 Vapor degrease

Vapor degrease the parts by lowering into the vapors. Hold the parts until vapors cease to condense on its surfaces.

3.3.1.2 Liquid degrease

Flush or circulate trichloroethylene at ambient temperature over or through the surfaces of the components for 10 to 30 minutes. Note safety precautions in paragraph 6.2 of this specification. For items requiring removal of varnish-type, organic material from the surface, immerse the part in acetone for 10 to 30 minutes.

NOTE: No open flame or smoking allowed due to its flammability.

3.3.1.3 Dry

Components shall be dried by circulating air at 180° F ($\pm 20^\circ$) 60 to 80 pounds per square inch through the part for 5 to 10 minutes. Alternate method of drying shall be accomplished with a drying oven or a vacuum oven.

3.3.1.4 Ultrasonic clean

Place the part in an ultrasonic bath consisting of 0.1 percent aqueous detergent solution (by volume).

3.3.1.5 Spray rinse

Spray rinse, flush, or immerse with distilled or demineralized water for 10 to 15 minutes at 90° F ($\pm 20^\circ$).

3.3.1.6 Dry

Components shall be dried using a gas meeting the requirements of paragraph 3.2.3 or in a vacuum oven of 180° F ($\pm 20^\circ$) at 5 to 50 mm of mercury for 1 hour. Items of grade 300 series stainless steel which have light corrosion at this point shall be processed as specified in paragraph 3.3.1.8. Heavily corroded areas shall be a cause for rejection of the part.

3.3.1.7 Process LOX service

Items which are to meet LOX cleanliness requirements shall be processed in accordance with paragraphs 3.3.1.7.1 and 3.3.1.7.2. Parts not intended for LOX service will not require these operations and shall be packaged and protected from contamination by wrapping with a minimum of two thicknesses of dry annealed aluminum foil secured with tape.

3.3.1.7.1 Degrease

Rinse, immerse, or circulate trichloroethylene over or through the surfaces for 2 to 5 minutes at ambient temperature. Note safety precautions given in paragraph 6.2 of this specification. Samples of the effluent shall be tested for nonvolatile residue and for particle size in the manner per Specification MSFC-SPEC-164.

3.3.1.7.2 Dry

Dry the components in the manner specified in paragraph 3.3.1.6 of this specification. Then package in the manners specified in paragraph 3.3.1.7.

3.3.1.8 Phosphoric acid

Parts made of Grade 300 series stainless steel which demonstrate corrosion after processing thru paragraph 3.3.1.6 shall be immersed in a dilute 10 to 20 percent solution of inhibited phosphoric acid at ambient temperature in an ultrasonic bath for 5 minutes. Rinse and dry the part thoroughly. The parts shall then be reprocessed from paragraph 3.3.1.3 through 3.3.1.7.

3.3.2 Process 2

Coated metallic components shall be cleaned by the following process (nonultrasonic).

3.3.2.1 Degrease

Carefully scrub the surfaces of the component using a nylon bristle brush, saturated with acetone or alcohol to remove any excess contamination.

3.3.2.2 Dry

Dry the part in the manner specified in paragraph 3.3.1.6 of this specification.

3.3.2.3 Wash

Scrub the surfaces of the component with a nylon brush saturated with five parts of Dowell F-33 detergent or equivalent, in 1000 parts demineralized water.

3.3.2.4 Rinse

Rinse the part for 10 to 15 minutes in demineralized water at 180° F ($\pm 20^\circ$).

3.3.2.5 Dry

Dry the part in the manner specified in paragraph 3.3.1.6 of this specification.

3.3.2.6 Process LOX service

Items which are to meet LOX cleanliness requirements shall be processed further in accordance with paragraph 3.3.1.7. Parts not intended for LOX service will not require these operations and shall be packaged in accordance with paragraph 3.3.1.7.

3.3.3 Process 3

Nonmetallic components, including metallic components combined with nonmetallic materials other than Teflon or Kel-F, shall be cleaned by the following process:

3.3.3.1 Wash

Immerse spray (1 inch nozzle) or flush each part in a solution of 5 parts (by volume) detergent to 1000 parts of demineralized water for 10 to 15 minutes at a temperature of 110°F ($\pm 10^{\circ}$). When it is feasible, the careful use of a nylon bristle brush may assist in removing contaminants.

3.3.3.2 Rinse

Rinse by immersing, flushing or spraying (1 inch nozzle) in demineralized water at 110°F ($\pm 10^{\circ}$) for 10 to 15 minutes. Handle with clean rubber gloves at all times during the cleaning operation.

3.3.3.3 Dry

Dry the part by drying in an oven or with missile grade air at 110°F ($\pm 10^{\circ}$) for 20 to 30 minutes and package in the manner specified in paragraph 3.3.1.7.

3.3.4 Process 4

Miscellaneous components.

3.3.4.1 Cleaning

Cleaning of continuous level probes for LOX service.

A. General - These probes are calibrated in the media being used; i.e. probes for fuel tanks are calibrated in fuel and probes for LOX tanks are calibrated in LOX.

B. Degrease - Place probes on rack in vapor degreasing tank and spray with trichloroethylene without lowering the rack into the degreasing tank.

C. Trichlorethylene - Circulate trichloroethylene through probe tubes and flush with clean trichloroethylene until the particle count is met and then sample for nonvolatile residue.

D. Dry - Subject the tubes to an oven at 125°F until dry.

3.3.4.2 Cleaning

Cleaning of continuous level probes for fuel service.

- A. Refer to paragraph 3.3.4.1B.
- B. Flush - Flush with deionized water until the particle count is met.
- C. Dry - Subject the tubes to an oven at 125° F until dry.

3.4 Recleaning Procedure

If additional cleaning, or recleaning, of Miscellaneous Components become necessary, they must be cleaned to the original cleanliness level by following the procedures outlined below.

3.4.1 Process 1

- If component is for LOX system, follow paragraphs 3.3.1.7.A and 3.3.1.7.B.

- If component is for a system other than LOX, follow paragraphs 3.3.1.4 and 3.3.1.5, then dry by using a gas meeting the requirements of paragraph 3.2.3 or in a vacuum oven of 180° F ($\pm 20^\circ$) at 5 to 50 mm of mercury for 1 hour.

3.4.2 Process 2

- If component is for LOX system, follow paragraphs 3.3.1.7.1 and 3.3.1.7.2.

- If component is for a system other than LOX, and has been previously cleaned as per paragraphs 3.3.2.1 through 3.3.2.5, repeat the same procedure outlined in paragraphs 3.3.2.1 through 3.3.2.5.

3.4.3 Process 3

To reclean components that were cleaned originally per Process 3, it will be necessary to repeat the process as outlined in paragraph 3.3.3.

3.4.4 Process 4

To reclean components that were cleaned originally per Process 4, it will be necessary to repeat the process as outlined in paragraph 3.3.4.

4. QUALITY ASSURANCE PROVISIONS

4.1 Acceptance Inspections

4.1.1 Visual inspections

The surfaces of all cleaned miscellaneous components intended for contact with LOX or GOX shall be visually inspected for the presence of moisture and foreign materials such as corrosion, scale, dirt, oil, grease, and similar foreign materials. The necessary special equipment such as a borescope shall be utilized to assure that internal surfaces are actually clean. The presence of visible contamination shall be cause for recleaning of the component by the LOX cleaning process.

4.1.2 Surface acidity or alkalinity

The surfaces of cleaned miscellaneous components shall be tested with pH indicating paper while the component is still wet from the last rinse, or after wetting the test surface with a few drops of distilled or demineralized water, to determine that the surface acidity or alkalinity registers a pH between 6.0 and 8.0. The pH indicating paper used shall be sensitive to the requirement specified above.

4.2 Reliability Tests

4.2.1 Particle size

Test solution for particle size analysis for miscellaneous components for use in oxygen, fuel, and pneumatic systems shall be prepared as specified in paragraph 4.4.1 and tested as specified in paragraph 4.4.2.1 of Specification MSFC-SPEC-164.

4.2.2 Nonvolatile residue content

Test solution for nonvolatile residue analysis for miscellaneous components for use in oxygen and pneumatic ground support systems shall be prepared as specified in paragraph 4.4.1 and tested as specified in paragraph 4.4.2.2 of Specification MSFC-SPEC-164.

5. PREPARATION FOR DELIVERY

5.1 Preservation and Packaging

Miscellaneous components shall be packaged immediately upon com-

pletion of the cleaning and drying processes in order to prevent recontamination. All openings into the component as well as surfaces intended for service in contact with LOX or GOX shall be protected by covering these areas with a minimum of two layers of aluminum foil reinforced with tape. The aluminum foil protected miscellaneous components shall then be packed in containers capable of providing protection against mechanical damage or recontamination during necessary storage, handling, or transportation before installation.

5.2 Marking

Cleaned miscellaneous components shall be marked with tags conforming to Type C, Specification UU-T-81F(1), which shall contain the following information:

- Part of identification number
- Method of cleaning and micron level
- Date of cleaning
- Title, date and number of this manufacturing specification
- Contractor identification
- Manufacturer's serial number
- Service medium or intended use (LOX, Fuel, Pneumatic service)

6. NOTES

6.1 Intended Use

This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use in the cleaning of miscellaneous components not covered by another cleaning specification.

6.2 Safety Precaution

Perform the trichloroethylene rinse or spraying operations under a fume exhaust hood or with a room exhaust system. Use of gloves is mandatory in all trichloroethylene operations. Use respirator, if no fume hood or room exhaust system is available.

NOTICE: When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING PROCESS DATA
CARBON REMOVAL FROM F-1 HEATING EXCHANGERS

1. SCOPE

1.1 Scope - This manufacturing procedure covers the approved Process Engineering Laboratory requirements for the carbon removal from the shell side of F-1 heat exchangers.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Vapor degreaser
- Two each mild steel tanks, air agitated, ventilated, capable of 175-210°F
- Two each mild steel rinse tanks, air agitated, ambient temperature
- One each stainless steel lined tank, mechanically agitated, capable of 260-280°F., ventilated
- One each Duracore lined tank, air agitated, ambient temperature, ventilated
- One each Plastisol lined rinse tank, air agitated, ambient temperature
- Air-water rinse gun
- Handling fixtures
- Hoist
- High pressure nozzles

2.2 Materials

- Trichloroethylene -- O-T-634
- Alkaline rust remover -- Cee Bee M-X-12 or Turco 4181
- Alkaline permanganate -- Cee Bee J-88 or Turco 4338
Scale conditioner
- Nitric acid -- O-A-88
- Water
- Compressed air

3. PROCEDURE

3.1 Preliminary Cleaning - Vapor degrease by slowly lowering the heat exchanger into trichloroethylene vapors. When condensation ceases to form, remove the heat exchanger slowly from the vapors.

3.2 Carbon Removal

3.2.1 Immerse in the alkaline rust remover solution (2 to 4 lb/gal) at 180-210°F. for a maximum of 60 minutes.

3.2.2 Rinse at ambient temperature by immersion, then use high pressure spray at approximately 150 psi by direct impingement on the carbon deposit areas to soften and remove the carbon.

3.2.3 Immerse in the alkaline permanganate solution (2.5 ± 0.3 lb/gal) at 175-200°F. for a maximum of 60 minutes. Spray rinse with an air-water gun during removal from the tank until the coloring is removed. Rinse by immersion in ambient temperature water.

NOTE: If carbon deposits covering more than 5 percent of the total area are present, repeat the above removal procedure until the carbon deposits are within the stated tolerances.

3.2.4 Passivate with 30 percent HNO_3 at ambient temperature for 30 to 60 minutes.

3.2.5 Rinse by immersing in ambient temperature water for 15 to 30 minutes.

3.2.6 Allow the heat exchanger to dry. Clean, dry compressed air may be used to accelerate the drying.

4. CONTROL

4.1 After cleaning, the heat exchanger shall be visually inspected for loose carbonaceous deposits. Presence of such deposits shall necessitate recleaning the part. Small tightly adherent deposits shall be permitted provided they do not cover more than 5 percent of the total area. Metal discolorations are permissible.

MANUFACTURING SPECIFICATION FOR
CLEANING OF UNLINED STAINLESS STEEL
FLEXIBLE HOSE

1. SCOPE

1.1 Scope — This specification designates the approved PE Laboratory manufacturing requirements for attaining the cleanliness levels for unlined stainless steel flexible hose, 2 inches or less in diameter, used in space launch vehicle applications.

1.2 Classification — Cleaning procedures set forth in this specification shall be applicable to the following grades of stainless steel; 304, 316, 321 and 347.

2. APPLICABLE DOCUMENTS

2.1 Governmental — The following documents, of the issue in effect on date of use of this specification, form a part of this specification.

SPECIFICATIONS

Federal

O-P-94a	Paper, Test; pH Indicator
O-T-634a	Trichloroethylene, Technical
BB-N-411a	Nitrogen
UU-T-81F	Tags, Shipping and Stock
PPP-T-60(3)	Tape, Pressure Sensitive Adhesive

Military

MIL-A-00148C(2)	Aluminum Foil, Dry Annealed
MIL-D-16791C	Detergents, Nonionic

George C. Marshall Space Flight Center

MSFC-SPEC-164

Cleanliness of Components for Use in
Oxygen, Fuel, and Pneumatic Systems,
Specification for

2.2 Other Publications - The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

Dow Chemical Company

Dowell F-33

Detergent

(Copies of the publication listed above may be obtained from Dowell Division, Dow Chemical Company, 1579 E. 21st Street, Tulsa, Okla.)

3. REQUIREMENTS

3.1 General - No deviation from the requirements of this manufacturing specification shall be permitted without the prior written approval of the MSFC initiating activity.

3.2 Materials

3.2.1 Drying or preservation gas - The nitrogen or air employed for drying or preservation shall be filtered to a 100 micron level, possess an oil content not greater than 3 parts per million (by weight), and contain a maximum of 24 parts per million (of moisture).

3.2.2 Tags - Tags employed in marking of cleaned components shall conform to Style C of Specification UU-T-81F.

3.2.3 Detergent - The detergent used in ultrasonic cleaning of flexible hose must be nonionic and comply with Specification MIL-D-16791C. Dowell F-33 is currently approved for use.

3.2.4 Test solvent - Trichloroethylene used for testing the cleanliness of LOX components shall meet the requirements of Type I of Specification O-T-634a, except that the nonvolatile residue shall not be greater than 0.010 gram per 500 ml at 221°F ($\pm 9^\circ$) when tested in accordance with paragraph 4.4.2.2 of Specification MSFC-SPEC-164. There shall be no particle over 175 microns in any dimension and no more than five particles from 100 to 175 microns in any dimension.

3.2.5 Liquid cleaning solvent - The liquid cleaning trichloroethylene used in this process shall meet the requirements of Specification O-T-634a, Type I.

3.2.6 pH indicator - The pH indicating paper used in this process for checking neutrality shall be of the medium range, Class A of Specification O-P-94a.

3.2.7 Tape - The tape used to secure and reinforce protective covers on cleaned hoses shall conform to Specification PPP-T-60.

3.2.8 Aluminum foil - The dry annealed aluminum foil used in this process for wrapping hoses shall comply with Specification MIL-A-00148C(2).

3.3 Procedure

3.3.1 Inspection - Visually inspect each component to determine its cleanliness and record any unusual contamination such as corrosion. If the part is corroded externally or internally, do not clean. Serialize each part rejected for corrosion or contamination.

3.3.2 Degrease - Rinse internally with trichloroethylene, meeting the requirements of paragraph 3.2.5 of this specification, at ambient temperature for 2 to 5 minutes and a pressure of 20 to 30 psi at the nozzle. Use respirator and gloves if any vapors are expelled into the atmosphere.

3.3.3 Drying - The flexible hose shall be dried by circulating preheated filtered air or nitrogen at 180°F ($\pm 20^\circ$) at 15 to 20 psi around and through the hose for 3 to 5 minutes.

3.3.4 Oven dry - Place the component in an oven at 180°F ($\pm 20^\circ$) for a minimum of 20 minutes or until free of trichloroethylene.

3.3.5 Ultrasonic clean - Immerse the component for 5 to 10 minutes in an ultrasonic cleaning bath containing 5 parts detergent (by volume) to 1000 parts demineralized water (by volume). Next, insert a power-driven, nylon brush revolving at 500 (± 50) revolutions per minute into the flexible hose while it is immersed, and continue brushing the hose interior for 3 to 5 minutes. As a precaution all metal parts of the brush or its driving shaft should be covered with tygon to prevent marring or scratching. The brushing must be carefully performed to avoid broken bristles lodging in the interior of the hose.

3.3.6 Rinse - Rinse the flexible hose by immersing, flushing, or spraying with demineralized water at ambient temperature for 5 to 10 minutes. Rinse, a second time, by passing a spray nozzle protected with teflon bumpers through the component with demineralized water at ambient temperature and at 50 to 60 psi pressure for 5 to 10 minutes. The pH of the effluent shall be in the range of 6 to 8.

3.3.7 Drying - Using air or nitrogen, meeting the requirements of paragraph 3.2.1 of this specification, with a pressure of 15 to 20 pounds per square inch and at 180°F ($\pm 20^\circ$), pressure dry the hose for 5 to 10 minutes.

3.3.8 Vacuum-oven dry - The operating temperature for vacuum-oven drying shall be 180°F ($\pm 20^\circ$). The vacuum shall be 740 mm of mercury and shall exist for 1 hour at temperature. Atmospheric pressure shall be restored by admitting air meeting paragraph 3.2.1 of this specification.

3.3.9 Inspect - Visually inspect the flexible hose for cleanliness and thorough dryness. Repeat operations in paragraphs 3.3.2 to 3.3.8 of this specification, if necessary to obtain clean, dry, flexible hose.

3.4 Recleaning Procedures - If additional cleaning, or recleaning, of unlined stainless steel flexible hose becomes necessary, they must be cleaned to the original cleanliness level by following the procedure outlined below.

3.4.1 Operation - Circulate prefiltered trichloroethylene through hose for 2 to 5 minutes and at a pressure of 20 to 30 psi. Obtain 500 ml sample from the effluent side of the system for particle and NVR determinations. Repeat procedure until limitations of Specification MSFC-SPEC-164 have been met.

3.4.2 Redry - When samples have tested satisfactorily, redry the hose per paragraph 3.3.7 of this specification, and repeat the vacuum-oven drying per paragraph 3.3.8 of this specification.

3.4.3 Package - Pack per paragraph 5.1 after recleaning.

4. QUALITY ASSURANCE PROVISIONS

4.1 Cleanliness - Flexible hose cleaned by this procedure shall meet the requirements of Specification MSFC-SPEC-164.

4.1.1 Reliability sample - Trichloroethylene which has been filtered through a Whatman 42 filter and which meets the low residue requirements of paragraph 3.2.4 of this specification shall be used for testing the cleanliness of samples taken from the production lot. Paragraphs 4.4.11 and 4.4.12 of Specification MSFC-SPEC-164, which relate to testing a square foot of surface area, shall govern the procedure with the further requirements that the hose shall be plugged with LOX-clean, stainless steel plugs and poured three-quarters full with the test liquid. The hose is then to be flexed thru a radius no greater than the minimum bend radius for a period of 1 minute. Repeat the flexing operation for a second and a third time. Empty the test liquid into a LOX-clean, container and cover tightly with aluminum foil. Test the liquid for particle size and nonvolatile residue per paragraphs 4.4.2.1 and 4.4.2.2 of Specification MSFC-SPEC-164. An alternate method requires circulating prefiltered trichloroethylene through the hose at ambient temperature and samples taken from the effluent side of the circulating system for particle and NVR determinations. Perform both operations above in a controlled atmosphere room during the entire process. Use of respirators (if 100 ppm trichloroethylene vapors are exceeded) and gloves are mandatory during these operations.

NOTE

Perform this operation under a fume exhaust hood, or use room exhaust system. Use gloves and respirator if no exhaust system is available.

4.2.2 Redry - When samples have tested satisfactorily, redry the hose per paragraph 3.3.7 of this specification, and repeat the vacuum-oven drying per paragraph 3.3.8 of this specification.

5. PREPARATION FOR DELIVERY

5.1 Preservation and Packaging - All metal flexible hose assemblies shall be protected by sealing all openings with approved secured closures, placing the assemblies in polyethylene bags, purging bags with a dry, oil-free gas to assure an inert package during storage, heat sealing both ends of bags, and packing as necessary to prevent recontamination or damage during handling and storage. Do not package any hose in the above manner if they are to be installed on vehicle soon after completion of cleaning cycle. Package these by wrapping ends with two layers of aluminum foil and secure the foil with tape.

5.2 Marking - Cleaned flexible hose shall be identified with tags meeting Specification UU-T-8F, Style C. The tags shall bear the following information:

- Part or serial number
- Method of cleaning, NVR and micron level (contractor)
- Date of cleaning
- Any information required by contract
- Manufacturer's serial number
- Intended use of component

6. NOTES

6.1 Intended Use - This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use in the cleaning of stainless steel, flexible hose for space launch vehicles.

NOTICE

When government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related government procurement operation, the United States Government incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING PROCESS DATA
CLEANING STAINLESS STEEL TUBING FOR INDUCTION BRAZING

1. SCOPE

1.1 Scope - This procedure describes an approved method for cleaning stainless steel tubing before induction brazing.

NOTE: This MPD does not include procedures for cleaning stainless steel tubing attached to instruments. These procedures are described in MPD 26302, entitled, "Cleaning Stainless Steel Tubing Attached to Instruments for Induction Brazing."

1.2 This procedure references the following documents:

20M84547	Glass Bead Peening
MSFC-SPEC-164	Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic Systems
MSFC-SPEC-237A	Solvent Precision Cleaning Agent
MSFC-PROC-404	Gases, Drying and Preservation, Cleanliness Level and Inspection Methods
MIL-G-994A	Glass Beads for Cleaning and Peening
AMS 2430E	Shot Peening

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Solvent Degreaser
- Cleaning Tanks
- Passivating Tanks

2.2 Materials

- Trichloroethylene
- Missile Grade Air, Hot
- Trisodium phosphate solution
- Deionized water
- Nitric Acid - Sodium Dichromate Solution
- Glass peening beads MIL-G-9954A
- Acetone, reagent grade
- Freon PCA

3. PROCEDURE

- 3.1 Solvent Degrease - Flush critical surfaces of components with solvent using pump on vapor degreaser.
- 3.2 Dry - Blow with drying gas (missile grade air per MSFC-PROC-404 or approved equivalent) at 160 to 180°F until dry.
- 3.3 Alkaline Cleaning - Clean with 10 percent solution of trisodium phosphate at 170 to 190°F by immersion or circulation for 10 to 15 minutes.
- 3.4 Rinse - Flush for 5 to 10 minutes with hot (160 to 180°F) deionized water.
- 3.5 Stainless Steel Passivation - Passivate with a solution of 20 ± 2 percent nitric acid and 2.0 ± 0.5 percent sodium dichromate at 120 to 130°F for 15 to 20 minutes by immersion or circulation.
- 3.6 Rinse - Flush with deionized water until the pH is in the 6 to 8 range.
- 3.7 Dry - Blow with drying gas (missile grade air per MSFC-PROC-404 or approved equivalent) at 160 to 180°F until dry.
- 3.8 Cleanliness Verification - Flush inside of tube with 500 ml sample of Freon PCA per MSFC-SPEC-237 until the Freon sample contains no particles greater than 175 microns.
- 3.9 Protection of Tube Interiors - Insert Teflon stoppers into the tube ends.

3.10 Peening - Glass bead peen a minimum of 1 inch length on the outside of the tube end to be brazed by directing a low pressure stream (30 to 40 psi) of glass beads on the tube until a clean matte finish is obtained.

3.11 Rinse - Leaving the Teflon stoppers in the tube ends, flush with cold water for 5 to 10 minutes followed by a flush with hot water at 160 to 180°F for 5 to 10 minutes.

3.12 Dry - Blow with drying gas (missile grade air per MSFC-PROC-404 or approved equivalent) at 160 to 180°F until dry.

3.13 Clean tube ends - Ultrasonically clean the tube ends in Freon PCA for 10 minutes at room temperature.

3.14 Complete unmasking - Remove the Teflon stoppers.

3.15 Cleaning tubes - Flush or spray tubes with Freon PCA to MSFC-SPEC-164, LOX.

3.16 Dry - Blow with drying gas (missile grade air per MSFC-PROC-404 or approved equivalent) at 160 to 180°F until dry.

3.17 Packaging

- Insert the tube in a Nylon bag and seal it.
- Install a plastic cap on each cleaned end of the tube over the Nylon film.
- Insert the bagged tube in a polyethylene bag and seal.
- The identification tag is to be placed inside the outer bag prior to sealing.

NOTE: A convenient number of tubes cleaned for brazing may be packaged in one bag without plastic caps with the individual tubes separated by a single heat seal across the bag.

MANUFACTURING PROCESS DATA FOR
CLEANING SS TUBING ATTACHED TO INSTRUMENTS FOR
INDUCTION BRAZING - ATM-ECS SYSTEM

1. SCOPE

1.1 Scope - This procedure describes an approved method for cleaning stainless steel tubing that is attached to instruments for induction brazing. This is a special case for the cleaning of stainless steel tubing for induction brazing (see MPD 26301C).

2. The following documents have been used in the preparation.

MSFC-DWG-20M84547	Glass Bead Peening
MIL-G-994A	Glass Beads for Cleaning and Peening
AMS 2430E	Shot Peening
MSFC-SPEC-164 (Amdt 5)	Specification Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic systems
MSFC-SPEC-237A	Solvent, Precision Cleaning Agent
MSFC-PROC-404	Procedure Gases, Drying, Preservation, Cleanliness Level and Inspection

3. EQUIPMENT AND MATERIALS

3.1 Equipment

3.1.1 Small nozzled probe for flushing with Freon PCA

3.1.2 Glass bead peening cabinet equipment

- 3.1.3 Trichloroethylene vapor degreaser
- 3.1.4 Ultrasonic cleaning tanks
- 3.2 Materials
 - 3.2.1 Teflon stoppers
 - 3.2.2 Nylon packaging film
 - 3.2.3 Polyethylene packaging film
 - 3.2.4 Tape for sealing
 - 3.2.5 Plastic protective caps
 - 3.2.6 Freon PCA per MSFC-SPEC-237
 - 3.2.7 Glass beads for peening MIL-G-994A
 - 3.2.8 Trichloroethylene
- 4. PROCEDURE

NOTE: Wear clean lint-free nylon gloves when performing these operations.

- 4.1 External Cleaning - Clean the external surfaces of the instrument by vacuuming and/or wiping it with a lint-free cloth slightly moistened with Freon PCA.
- 4.2 Preparation for Peening
 - 4.2.1 Protection of tube interiors - Insert clean Teflon stoppers into the open tube ends carefully. Avoid damaging delicate parts or diaphragms.
 - 4.2.2 Protection of instrument bodies - Wrap the body of the instrument carefully in clean nylon plastic film, leaving exposed about 1 or 2 inches of the tube ends that are to be induction brazed.

Fasten the film wrapping around the exposed tube ends with rubber bands or tape. Do not permit the tape adhesive to touch the tube surface.

4.2.3 Solvent degrease - Flush the critical external surfaces of the tubes with trichloroethylene solvent using the hose in the vapor degreaser.

4.2.4 Dry - Blow hot missile grade air at 160° to 180°F across the surface of the exposed tubing ends until they are dry.

4.3 Peening Operations

4.3.1 Peening tube ends to be brazed - Peen the surface of the tube ends to be brazed for a distance of approximately 1 inch from the end by directing a stream of glass beads at low pressure (30 to 40 psi), away from the instrument, towards the end of the tube, until a clean matte finish is obtained.

4.3.2 Rinse - Flush the peened tube surfaces with cold water for 5 to 10 minutes followed by a hot water flush at 160° to 180°F for 5 to 10 minutes.

4.3.3 Dry - Blow hot missile grade air at 160° to 180°F across the surface of the tube ends until they are dry.

4.3.4 Unpackage - Remove the nylon packaging film protecting the instrument body.

4.4 Final Cleaning

4.4.1 Ultra cleaning tube ends - Ultrasonically clean the tube ends in Freon PCA for 10 minutes at room temperature.

4.4.2 Dry - Blow hot missile grade air at 160° to 180°F across the surface of the tube ends until they are dry.

4.4.3 Clean body - Clean the body of the instrument by wiping it carefully with clean lint-free wipes moistened with Freon PCA until it is visually clean.

4.4.4 Remove stoppers - Carefully remove the Teflon stoppers from the tube ends.

4.5 Final Testing - There are two types of instruments each requiring different cleaning and testing methods.

1. Flow Thru Type.
2. Dead End Type.

4.5.1 Testing flow through type - For the instruments with flow thru tubing, Flush the inside of the tubing with Freon PCA until a 500 ml sample of the effluent contains no particulate contamination greater than 175 microns.

4.5.2 Testing dead end type - For the instruments with dead end tubing, Flush the inside of the tubing with Freon PCA using the small nozzle probe to direct the stream well up into the tubing; until a 500 ml sample of the effluent contains no particulate contamination greater than 175 microns.

4.6 Final Packaging

4.6.1 Carefully insert the cleaned instrument assembly into a clean Nylon 6 bag and seal it. Place the plastic protective caps over the tube ends. The nylon film will be between the tube and the cap. -

4.6.2 Carefully insert the nylon bagged assembly into a clean polyethylene outer bag being careful not to dislodge the plastic cap covers over the tube ends. Place the identification tag inside the polyethylene bag, so that it is visible through it, and seal the outer polyethylene bag.

The instrument is ready for shipment.

MANUFACTURING SPECIFICATION FOR
CLEANING OF UNLINED STAINLESS STEEL BELLOWS

1. SCOPE

1.1 Scope - This specification covers the approved PE Laboratory manufacturing requirements for attaining the cleanliness standards for unlined stainless steel bellows used on space launch vehicles.

1.2 Applicability

1.2.1 Stainless steel grades - Cleaning requirements set forth in this specification shall be applicable to bellows made of the following grades of stainless steel; 304, 316, 321, and 347.

1.2.2 Types - This specification may apply to stainless steel bellows joined to aluminum flanges, as well as to those consisting only of stainless steel, provided the aluminum is protected during processing. This specification does not apply to bellows which are not sealed by cap welding, or any bellows that cannot be positively dried by vacuum-oven drying.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this specification, form a part of this specification.

SPECIFICATIONS

Federal

O-A-88(1)	Acid, Nitric, Technical Grade
O-H-795	Acid Hydrofluoric, Technical Grade
O-P-94a	Paper, test; pH Indicator
O-T-634a	Trichloroethylene, Technical
BB-N-411a	Nitrogen

UU-T-81d

Tags, Shipping and Stock

PPP-T-60(2)

Tape, Pressure Sensitive Adhesive,
Waterproof for Packaging and Sealing

Military

MIL-A-00148(ORD)

Aluminum Foil, Dry Annealed

George C. Marshall Space Flight Center

MSFC-SPEC-164

Cleanliness of Components for use in
Liquid Oxygen Fuel and Pneumatic
Systems

MS 150.0

Manufacturing Specification for the
Vapor Degreasing of Metallic Surfaces

2.2 Other Publications - The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

TURCO PRODUCTS, INC.

Tech. Product Data

Turco Vitro-Klene

Bulletin No. 76

(Copies of the publication listed above may be obtained from Turco Products, Inc., 6135 S. Central Avenue, Los Angeles 1, California.)

FIDELITY CHEMICAL PRODUCTS CORP.

Plastic Coating Compound

Strippable Hot Dip Type

(Copies of the publication listed above may be obtained from the Fidelity Chemical Products Corporation, Newark, New Jersey.)

3. REQUIREMENTS

3.1 General - No deviation from the requirements of this manufacturing specification shall be permitted without the prior written approval of the MSFC initiating activity.

3.2 Materials

3.2.1 Vapor degreasing solvent - Trichloroethylene used in this process shall meet the requirements of Type II of Specification O-T-634a.

3.2.2 Test solvent - Trichloroethylene used for testing the cleanliness of LOX components shall meet the requirements of Type I of Specification O-T-634a, except that the nonvolatile residue shall not be greater than 0.010 gm. per 500 ml when the solution is heated to 221 to 230°F and that no particle larger than 175 microns in any dimension shall be present.

3.2.3 Demineralized water - Demineralized water shall be filtered to remove all particles larger than 175 microns and shall possess a minimum electrical resistivity of 50 000 ohm.

3.2.4 Alkaline cleaner - The alkaline cleaner used for degreasing of metals in this process shall be equal in quality of cleaning to Turco Vitro-Klene.

3.2.5 Cellulose acetate - The cellulose acetate employed for protecting metals or areas from attack by the chemicals employed in this process shall be equal in quality to Fidelity Chemical Products Corporation, Plastic Coating Compound.

3.2.6 Aluminum foil - Dry-annealed aluminum foil employed in this process for protecting cleaned bellows against contamination shall conform to paragraph 6.3.2 of Specification MIL-A-00148 (ORD).

3.2.7 Tags - Tags employed in marking of cleaned components shall conform to style C of Specification UU-T-81d.

3.2.8 pH Indicator - The pH indicating paper used in this process for checking neutrality shall be of the medium range, Class A, of Specification O-P-94a.

3.2.9 Drying and preservation gases - The nitrogen or air employed for drying or preservation shall be filtered to a 100 micron level, possess an oil content not greater than 3 parts per million (by weight), and contain a maximum of 24 parts per million (by volume) of moisture.

3.3 Drying

3.3.1 Oven drying - When drying is performed in an oven, the operating temperature shall be 180°F (±20°) and the oven shall be purged with gas meeting the requirements of paragraph 3.2.9 of this specification.

3.3.2 Vacuum-oven drying - The operating temperature for vacuum-oven drying shall be 180°F (±20°). The vacuum, as read on the alphanon gage, shall be 740 mm of mercury and shall exist for 1 hour at temperature. Atmospheric pressure shall be restored by admitting air meeting paragraph 3.2.9 of this specification.

3.3.3 Compressed, heated gases - When drying is performed by blasting or purging a component with heated, compressed gases, the pressure shall be 60 (±20) psi and the temperature shall be 180°F (±20°). The gas shall conform to paragraph 3.2.9 of this specification.

3.4 Procedure

3.4.1 Flanged stainless steel bellows - This procedure covers unlined bellows or subassemblies made only of stainless steel.

3.4.1.1 Inspection - Visually inspect each part to determine its cleanliness and record any unusual contamination.

3.4.1.2 Vapor degrease - Vapor degrease per MS 150.0.

3.4.1.3 Hot alkaline clean - Immerse in an aqueous solution of Turco Vitro-Klene heated to 200°F (±5°) for 10 to 15 minutes. Vitro-Klene concentration in solution shall be 8 to 10 ounces per gallon of water.

3.4.1.4 Hot water rinse - Rinse in hot water at 180°F (±10°).

3.4.1.5 Spray - Spray rinse in cold water for 3 to 5 minutes.

3.4.1.6 Pickle - Immerse each unit for 20 to 30 minutes in an acid solution of 20 (± 5) percent nitric acid (by weight) and 1.5 (± 0.5) percent hydrofluoric acid (by weight) operating at 150°F ($\pm 10^\circ$).

3.4.1.7 Rinse - Rinse in tank with overflowing water for 10 to 15 minutes at ambient temperature.

3.4.1.8 Passivate - Immerse each unit for 15 to 20 minutes in a 20 (± 5) percent nitric acid (by volume) and 2 (± 0.5) percent potassium dichromate (by weight) solution at 125°F ($\pm 5^\circ$).

3.4.1.9 Rinse - Rinse in demineralized water for 15 to 20 minutes at ambient temperature.

3.4.1.10 Spray - Spray rinse with demineralized water for 3 to 5 minutes at ambient temperature until a pH of 6 to 8 is reached.

3.4.1.11 Dry - Dry with pressurized hot gas per paragraph 3.3.3 of this specification.

3.4.1.12 Oven - Oven dry per paragraph 3.3.1 of this specification.

3.4.1.13 Degrease - Rinse each unit for at least 5 minutes with trichloroethylene suitable as a test liquid which is covered in paragraph 3.2.4 of this specification. Repeat this rinse for two more rinsings. Sample 500 cm³ of effluent into LOX clean containers and analyze per paragraph 4.4.2 of Specification MSFC-SPEC-164 for nonvolatile residue and particle size.

NOTE: Perform this operation under a fume exhaust hood. Use gloves and respirator if a fume exhaust hood is not available.

3.4.1.14 Dry - Dry with pressurized hot gas per paragraph 3.3.3 of this specification.

3.4.1.15 Vacuum dry - Vacuum dry per paragraph 3.3.2 of this specification.

3.4.1.16 Inspect - Check bellows visually for cleanliness and thorough dryness.

3.4.1.17 Preserve and package - Bellows should be protected and packaged per Specification MSFC-SPEC-164.

3.4.2 Aluminum flanged stainless steel bellows - This procedure applies to unlined stainless steel bellows with aluminum flanges.

3.4.2.1 Vapor degrease - Vapor degrease per Specification MS 150.0.

3.4.2.2 Quick-dip - Coat each aluminum flange or area desired to be protected with hot plastic coating compound by dip immersion at 350° F ($\pm 10^\circ$). Allow to cool and harden.

3.4.2.3 Hot alkaline clean - Immerse in an aqueous solution of Turco Vitro-Klene heated to 200° F ($\pm 5^\circ$) for 10 to 15 minutes. Vitro-Klene concentration in the solution should be from 8 to 10 ounces per gallon of water.

3.4.2.4 Hot water rinse - Rinse in hot water at 180° F ($\pm 10^\circ$).

3.4.2.5 Spray - Spray rinse in cold water for 3 to 5 minutes.

3.4.2.6 Pickle - Immerse each unit for 20 to 30 minutes in an acid solution of 20 (± 5) percent nitric acid (by weight) and 1.5 (± 0.5) percent hydrofluoric acid (by weight) operating at 150° F ($\pm 10^\circ$).

3.4.2.7 Rinse - Rinse in a tank with overflowing water for 10 to 15 minutes at ambient temperature.

3.4.2.8 Passivate - Immerse each unit for 15 to 20 minutes in a solution of 20 (± 5) percent nitric acid (by weight) and 2 (± 0.5) percent potassium dichromate (by weight) at 120° F (± 0.5) percent potassium dichromate (by weight) at 120° F ($\pm 5^\circ$).

3.4.2.9 Rinse - Rinse in demineralized water for 15 to 20 minutes at ambient temperature.

3.4.2.10 Spray - Spray rinse with demineralized water for 3 to 5 minutes at ambient temperature until a pH of 6 to 8 is attained.

3.4.2.11 Dry - Dry with pressurized hot gas per paragraph 3.3.3 of this specification.

3.4.2.12 Stripping - Completely strip the plastic coating compound off the flanges or other protected areas.

- 3.4.2.13 Rinse - Rinse the unit in demineralized water for 2 to 5 minutes at ambient temperature.
- 3.4.2.14 Dry - Dry with pressurized hot gas per paragraph 3.3.3 of this specification.
- 3.4.2.15 Oven - Vacuum dry per paragraph 3.3.1 of this specification.
- 3.4.2.16 Degrease - Rinse each unit for at least 5 minutes with trichloroethylene suitable as a test solvent and which is covered in paragraph 3.2.4 of this specification. Repeat this rinsing for two more rinsings. Sample 500 cm³ of the effluent into LOX clean containers and analyze per paragraph 4.4.2 of Specification MSFC-SPEC-164 for nonvolatile residue and particle size, (Refer to paragraph 3.4.1.13).
- 3.4.2.17 Dry - Dry with pressurized hot gas per paragraph 3.3.3 of this specification.
- 3.4.2.18 Vacuum dry - Vacuum dry per paragraph 3.3.2 of this specification.
- 3.4.2.19 Inspect - Check bellows visually for cleanliness and thorough dryness.
- 3.4.2.20 Preserve and package - Bellows should be protected and packaged per Specification MSFC-SPEC-164.
- 3.4.3 Recleaning - If the additional cleaning or recleaning of unlined stainless steel bellows becomes necessary, they must be cleaned to the original cleanliness level by following the procedure outlined below.
- 3.4.3.1 Operation - Follow paragraphs 3.4.2.16 through 3.4.2.20.

4. QUALITY ASSURANCE PROVISIONS

- 4.1 Level of Cleanliness - Components cleaned by this procedure shall meet the requirements of Specification MSFC-SPEC-164.
- 4.2 Solution Maintenance - Tank solutions employed in alkaline degreasing, pickling, and passivating shall be analyzed periodically to preserve efficiency by maintaining their compositions within operating limits.

4.3 Quality Control - Control of the quality of various tank operations can be assured by adherence to the time and temperature specified.

5. PREPARATION FOR DELIVERY

5.1 Preservation and Packing - The sealed, finished bellows shall be packaged and preserved in accordance with Specification MSFC-SPEC-164, paragraph 3.6.1.5.

5.2 Marking - When applicable, the tags attached to the sealed bellows shall record the following information.

- Part or identification number
- Contractor identification
- Method of cleaning and particle size, micron level
- Date of cleaning
- Service medium or intended use of component
- Any further information required by contract

6. NOTES

6.1 Intended Use - This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use in cleaning of unlined stainless steel bellows for space launch vehicle applications.

NOTICE: When government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related government procurement operation, the United States Government incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

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Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory

George C. Marshall Space Flight Center

MANUFACTURING SPECIFICATION FOR THE
INTERNAL PROTECTION OF STAINLESS STEEL PIPE
AND TUBING DURING FUSION WELDING

1. SCOPE

1.1 Scope - This specification covers the approved PE Laboratory manufacturing requirements for insuring adequate protection to the inside of stainless steel pipe and tubing during fusion welding of lap joints for space launch vehicle applications.

1.2 Applicability - The procedure outlined in this specification shall apply to all types and sizes of stainless steel pipe and tubing used in space launch vehicles when argon is used as the shielding gas.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this specification, form a part of this specification.

SPECIFICATIONS

Military

MIL-A-4144 (USAF) (1) Argon, Gas, Welding

2.2 Other Publications - The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

The Linde Company

Publication No. F808 Operating Instructions for the Hi-Vacuum
Bulb Tester

(Copies of the publication listed above may be obtained from the Linde Company, Division of Union Carbide Corporation, 270 Park Avenue, New York 17, New York.)

3. REQUIREMENTS

3.1 General - No deviation from the requirements of this manufacturing specification shall be permitted without prior written approval of the MSFC initiating activity.

3.2 Conflicts - In case of conflict between this specification and any other specification, standard or publication listed herein, this specification shall govern.

3.3 Personnel - Only qualified personnel shall determine the purity of inert atmospheres used for the internal protection of stainless steel pipe and tubing during welding.

3.3.1 Qualification - Qualification shall consist of instruction and testing in the use of oxygen analyzing equipment by the facility involved.

3.4 Equipment

3.4.1 General- All welding equipment shall be capable of producing satisfactory welds when operated by a certified welder, or by a welding operator using a qualified procedure.

3.4.2 Gas regulators and flow meters - Gas regulators and flow meters shall accurately meter flow rates of argon gas from 1 to 20 cubic feet per hour.

3.4.3 Oxygen gas analyzer - The instrument used for determining the oxygen content of an argon atmosphere, to meet the requirements of this specification, shall be a Linde Hi-Vacuum Bulb Tester, or equal.

3.4.4 Miscellaneous equipment - The tygon tubing, plugs, fittings, and valves used in connecting the test instrument to the work section shall be clean, dry, and noncontaminating to the flowing inert gas. All fittings shall be tight to prevent any air influx into the system.

3.5 Safety Precautions

3.5.1 The operator shall wear safety glasses during the operation of the test instrument.

- 3.5.2 The maximum pressure used within the test instrument shall be 5 psi.
- 3.5.3 A pressure regulator and a flow meter shall be installed in the line between the argon gas supply and the test instrument.
- 3.6 Protective Atmosphere Purity - The argon gas used as a protective atmosphere shall contain less than 20 parts per million of oxygen.
- 3.7 Atmosphere Production and Evaluation Procedure - The argon atmosphere used to protect the interior of work sections during fusion welding shall be produced and evaluated in accordance with the following procedure when using the Linde Hi-Vacuum Bulb Tester.
- 3.7.1 The two lengths of stainless steel pipe or tubing to be fusion welded shall be chemically or mechanically cleaned in preparation for welding.
- 3.7.2 The two lengths of stainless steel pipe or tubing shall be fitted together as shown in Figure 5-1.
- 3.7.3 Install two plugs, A and B, at points approximately 3 inches on each side of the intended weld joint (see Figure 5-1).
- 3.7.4 Connect a tested and approved argon gas supply (see paragraph 4.1) through a suitable pressure regulator and flow meter, to the inlet side of the chamber formed by plugs A and B (see Figure 5-1).
- 3.7.5 Connect a gas line from the outlet side of the chamber, formed by plugs A and B, to a Y connector (see Figure 5-1).
- 3.7.6 Connect a line from one leg of the Y connector to the outlet flow meter.
- 3.7.7 Connect a line from the second leg of the Y connector to the inlet fitting of the test instrument (see Figure 5-1).
- 3.7.8 Close the valve on the test instrument, open both valves on the Y connector, and open the valve on the outlet flow meter.

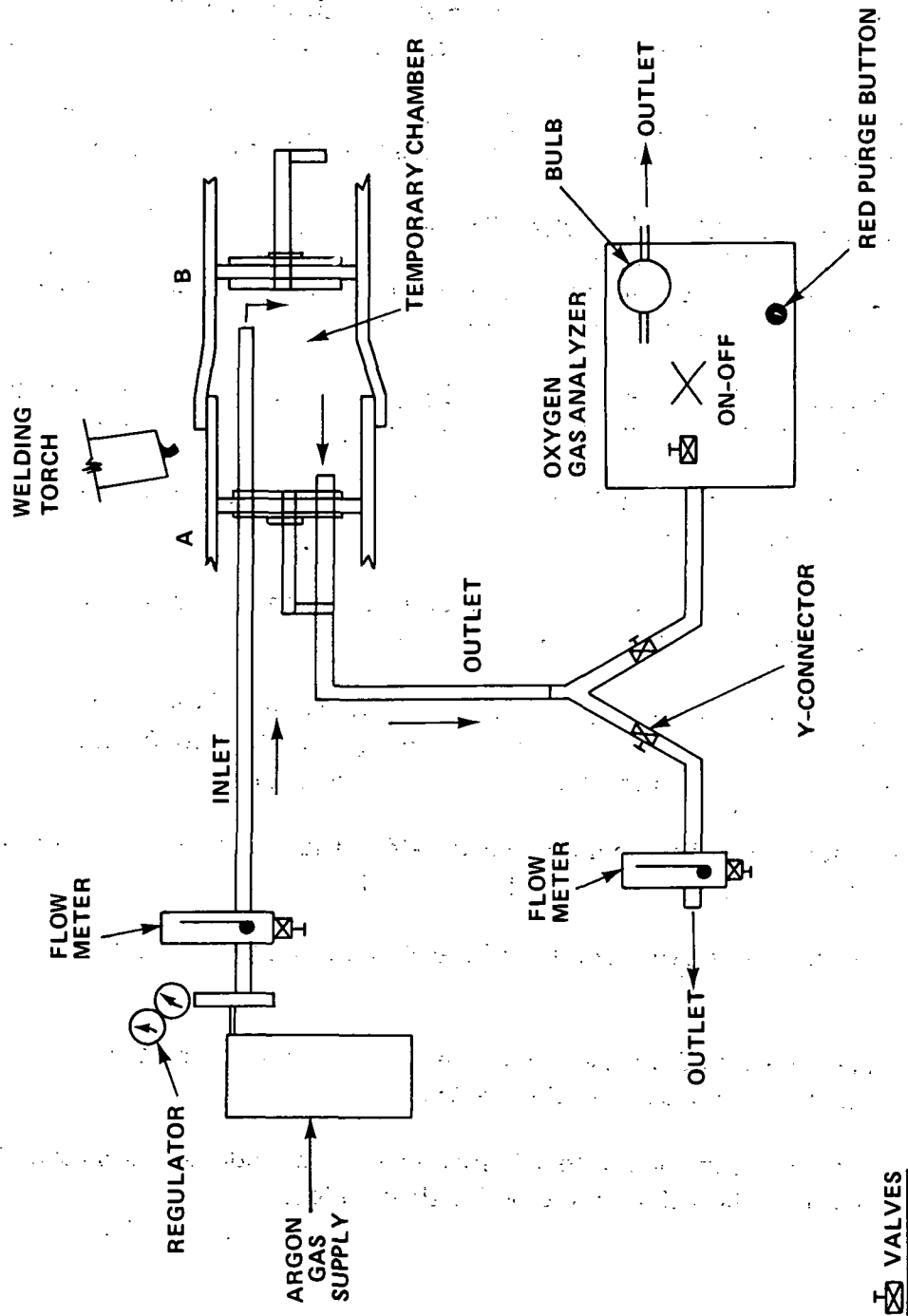


Figure 5-1. Schematic for insuring adequate protection to the inside of stainless steel tubing during welding.

3.7.9 Open the valve from the argon gas supply, set the flow meter on the inlet side of the system at 20 cubic feet per hour, and allow the inert gas to flow into and out of the chamber formed by plugs A and B.

3.7.9.1 A 15 and 35 minute purging time for a 4 inch and 8 inch tube respectively is adequate to reduce the oxygen within the chamber to less than 20 parts per million.

3.7.9.2 Other size tubes require an instrument check test as given in this section, every 5 minutes of inert gas flow, to determine when oxygen contamination is below the specified 20 parts per million.

3.7.10 Reset inlet flow meter so that the outlet flow meter shows a flow of 5 cubic feet per hour. Open valve on the test instrument and allow outlet gas from chamber to flow through the test instrument for 5 minutes.

3.7.11 Place switch of test instrument in the OFF position, plug electrical cord into a 110-volt, 60-cycle voltage source, set Variac to 85 volts, and clean filament by depressing the red PURGE BUTTON for approximately 1 second. The filament shall be in a clean, bright condition.

3.7.12 Allow the outlet gas from the chamber to flow through the test instrument for 5 additional minutes, set the Variac to 18 volts, turn the switch to the ON position for exactly 30 seconds, and then turn to OFF position.

3.7.13 Visually examine the filament and note its color. If the filament has a discoloration (See Table 1), oxygen contamination is at an unacceptable level. The test procedure (steps 3.7.9 through 3.7.14) must be repeated after a proper purge interval until the oxygen contamination is less than 20 parts per million.

3.7.14 If the tungsten filament has no discoloration, the tube is ready for welding. (See Table 5-1).

3.7.14.1 Reduce outlet gas flow from 1 to 2 cubic feet per hour to avoid blowing out of molten weld metal as it is deposited.

4. QUALITY ASSURANCE PROVISIONS

4.1 Argon Gas Quality - The following test shall be used to determine whether the quality of an argon gas supply is sufficiently high to allow it to meet the requirements of this specification.

4.1.1 Connect the argon gas supply to be tested, through a suitable pressure regulator and flow meter, to the inlet side of the test instrument.

4.1.2 Open the test instrument inlet valve.

4.1.3 Set the inlet pressure regulator so that the maximum pressure is 5 psi.

4.1.4 Set the flow meter so that the rate of flow through the test instrument at 5 psi is 5 cubic feet per hour.

4.1.5 The inert gas shall flow through the test instrument for 5 minutes.

4.1.6 Place switch in the OFF position, plug electrical cord into a 110-volt, 60-cycle voltage source, set Variac to 85 volts, and clean filament by depressing the red PURGE BUTTON for about 1 second.

4.1.6.1 If the bulb filament does not become clean and bright, the oxygen content is above the acceptability level as stated by this specification. Such a gas supply shall be rejected.

4.1.7 Allow argon gas from the cylinder or manifold to flow through the test instrument for an additional 5 minutes, set the Variac to 18 volts, turn the switch to the ON position for exactly 30 seconds, and then turn to the OFF position.

4.1.8 Visually examine the filament and note its color. Use data in Table 5-1 to determine oxygen content.

TABLE 5-1. FILAMENT COLOR AND OXYGEN CONTENT

Filament Color	Oxygen Content, ppm
Black	100 to 400

TABLE 5-1. (Concluded)

Tan	Approaching 40
Overall dullness with color at ends	Approaching 20
No discoloration	Less than 20

4.1.9 The acceptance value for an argon gas supply shall be based on an oxygen content of less than 20 parts per million as determined by the above test procedure.

5. PREPARATION FOR DELIVERY

Not applicable to this specification.

6. NOTES

6.1 Intended Use - This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use in the internal protection of stainless steel pipe and tubing during fusion welding for space launch vehicle applications.

NOTICE: When Government drawings, specifications or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

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MANUFACTURING SPECIFICATION FOR CLEANING AND PASSIVATION OF HEAT EXCHANGERS

1. SCOPE

1.1 Scope - This manufacturing specification covers the approved PE Laboratory requirements for the cleaning and passivation of heat exchanger assemblies and components for space launch vehicle application.

1.2 Applicability - The procedures set forth in this manufacturing specification are applicable to heat exchanger assemblies fabricated from types 304L, 321, and 347 austenitic stainless steels.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this specification, form a part of this specification.

SPECIFICATIONS

Federal

O-A-88(1)	Acid, Nitric, Technical Grade
O-C-303B	Chromium Trioxide, Technical (Chromic Acid)
O-H-795 (2)a	Hydrofluoric Acid, Technical
O-P-94A	Paper, Test, pH Indicator
O-S-595A(3)	Sodium Dichromate, Technical Grade
O-S-598	Sodium Hydroxide (Caustic Soda), Technical
O-T-634A	Trichloroethylene, Technical

STANDARDS

Military

MIL-STD-171(ORD)

Systems for Preparation, Painting
and Finishing Metal and Wood Surfaces

DRAWINGS

George C. Marshall Space Flight Center

10438000

Heat Exchanger Assembly (Weldment)

3. REQUIREMENTS

3.1 General - No deviation from the requirements of this manufacturing specification shall be permitted without prior written approval of the MSFC initiating activity.

3.2 Cleaning and Passivating Materials

3.2.1 Trichloroethylene for vapor degreasing - The trichloroethylene used for vapor degreasing under the provisions of this specification shall meet all of the requirements for Type II of Specification O-T-634a.

3.2.2 Trichloroethylene for cold degreasing - The trichloroethylene used for flushing or immersion degreasing under the provisions of this specification shall meet all of the requirements for Type I of Specification O-T-634a, except that the nonvolatile residue content shall be not greater than 0.010 gram per 500 mil when tested as specified in 4.4.2.2 of Specification MSFC-SPEC-164. No 500-ml sample shall contain a particle larger than 175 microns in any dimension or more than 5 particles between 100 and 175 microns in any dimension.

3.2.3 Nonionic detergent - The nonionic detergent used for cleaning under the provisions of this specification shall meet all of the requirements for Type II of Specification MIL-D-16791C. (The only detergent approved for use at this time is Dowell F-33.)

3.2.4 Demineralized water - The minimum specific resistance of demineralized water shall be 50000 ohm. It shall contain no particles larger than 175 microns in any dimension.

3. 2. 5 Drying or preservation gases - Gases used in drying or preserving stainless steel heat exchanger assemblies shall contain no particles larger than 100 microns (absolute), the oil content of the gases shall not be greater than 3 parts per million (by weight), and the moisture content of the gases shall be not greater than 24 parts per million (by volume).

3. 2. 5. 1 Nitrogen - Nitrogen gas, to be used in drying or preservation processes, shall conform to Type II, Class I, Grade B of Specification BB-N-411a, except that the particle size, oil content, and moisture content requirements of 3. 2. 5 shall be met.

3. 2. 5. 2 Compressed air - Compressed air used in drying or preservation processes shall conform to paragraph 3. 2. 5 above.

3. 2. 6 Nitric acid - Nitric acid, to be used in cleaning and passivating processes, shall be of technical grade conforming to Specification O-A-88(1).

3. 2. 7 Hydrofluoric acid - Hydrofluoric acid, to be used in cleaning processes, shall be of technical grade conforming to Specification O-H-795(2)a.

3. 2. 8 Sodium dichromate - Sodium dichromate for use in passivating stainless steel heat exchangers shall be of technical grade conforming to Specification O-S-595A(3).

3. 2. 9 Chromic acid - Chromic acid for use in the sealing of stainless steel heat exchangers shall be of technical grade conforming to Specification O-C-303B.

3. 2. 10 Paper, pH indicating - The pH indicating paper used in the checking of stainless steel heat exchangers shall be of a grade conforming to Specification O-P-94A.

3. 2. 11 Aluminum foil - The aluminum foil used in the protection of cleaned stainless steel heat exchangers shall conform to Specification MIL-A-00148B (ORD) and shall be free of oil and foreign matter.

3. 2. 12 Tape - The tape used to secure protective covers on cleaned stainless steel heat exchangers shall conform to Specification PPP-T-60(2).

3. 2. 13 Tags - The tags used to mark and identify cleaned heat exchanger assemblies shall conform to Specification UU-T-81D(1).

3.3 Cleaning of Stainless Steel Heat Exchanger Components Before Welding - Heat exchanger components, which are to be fusion welded into a heat exchanger assembly (Part No. 10438000), shall have both external and internal surfaces thoroughly degreased and the surface oxides mechanically removed for a distance of 1 inch on both sides of the weld location before welding.

3.4 External Cleaning of Stainless Steel Heat Exchanger Components After Fusion Welding - Mechanically clean all areas containing weld oxide or scale by means of a precleaned 300 series stainless steel wire brush immediately after the completion of the fusion welding operation.

3.5 Metal Conditioning After Heat Exchanger Fabrication

3.5.1 Install clean, blind flanges, and teflon gaskets on both the LOX inlet flange and the GOX outlet flange of the heat exchanger assembly.

3.5.2 Vapor degrease heat exchanger assembly at 188 °F ($\pm 3^\circ$) until vapors cease to condense on the assembly surface.

3.5.3 Air dry heat exchanger assembly.

3.5.4 Immerse the entire heat exchanger assembly in a solution containing 20 (± 2) percent nitric acid (by volume) and 2 (± 0.5) percent sodium dichromate (by weight) at 125 °F ($\pm 5^\circ$) for 30 to 60 minutes.

3.5.5 Rinse the entire heat exchanger assembly with demineralized water spray at ambient temperature for 10 (± 5) minutes.

3.5.6 Immerse the entire heat exchanger assembly in a solution containing 5 (± 0.5) percent sodium dichromate (by weight) at 150 °F ($\pm 10^\circ$) for 60 (± 5) minutes.

3.5.7 Rinse the heat exchanger in demineralized water at ambient temperature for 10 minutes.

3.5.8 Immerse the heat exchanger assembly in a demineralized water solution containing sufficient chromic acid to give a pH of 3 to 5, at 185 °F (± 25) for a minimum of 5 minutes.

3.5.9 Dry the heat exchanger assembly in an oven at 200° F ($\pm 20^\circ$) for a minimum of 1 hour.

3.5.10 Remove blind flanges and Teflon gaskets from the LOX inlet flange and the GOX outlet flange.

3.5.11 Seal openings of the LOX inlet flange, GOX outlet flange and both end openings in body of heat exchanger assembly with a minimum of two thicknesses of aluminum foil reinforced with tape.

3.6 Heat Exchanger Assembly - Cleaning of internal tube surfaces shall be pickled and passivated after final welding where no lap welds exist. Any repickling or repassivating of these tubes shall be done with the approval of the procuring activity.

3.6.1 Thoroughly degrease coils internally by circulating Type I trichloroethylene at ambient temperature through the coils at 15 to 25 psi for 5 to 10 minutes. Use respirators unless room exhaust is available as a safety precaution. Use of gloves is mandatory for this operation.

3.6.2 Thoroughly dry the degreased coils by blowing compressed air through them until all liquid trichloroethylene has evaporated. Use nitrogen or air at 180°F ($\pm 20^\circ$) at 15 to 25 psi pressure for 15 to 20 minutes.

3.6.3 Chemically clean the coils internally by circulating a solution of 10 (± 1) percent (by weight) of sodium hydroxide plus 5 parts (by volume) nonionic detergent in 1000 parts (by volume) of demineralized water through them at 180°F ($\pm 10^\circ$) and 60 (± 20) psi for 15 to 20 minutes.

3.6.4 Rinse the coils internally by circulating tap water through the coils at 170°F ($\pm 10^\circ$) and 60 (± 20) psi for 15 (± 5) minutes.

3.6.5 Chemically pickle the coils internally by circulating a solution of 20 (± 2) percent nitric acid (by weight) plus 1.5 (± 0.5) percent hydrofluoric acid (by weight) through the coils at 95°F ($\pm 5^\circ$) and 60 (± 20) psi for 5 to 8 minutes.

3.6.6 Rinse the coils internally by circulating tap water through the coils at 170°F ($\pm 10^\circ$) and 60 (± 20) psi for 15 (± 5) minutes.

3.6.7 Any tubing in the heat exchanger assembly with scale or contamination products remaining after the 5 minute pickling operation, must be cleaned internally by wire brushing with a 300 series stainless steel brush.

3.6.8 Passivate the coils internally by circulating a solution of 20 (± 2) percent nitric acid by volume plus 2 (± 0.5) percent sodium dichromate by weight through the coils at 125°F ($\pm 5^\circ$) and 60 (± 20) psi for 15 to 20 minutes.

3.6.9 Rinse the coils internally by circulating demineralized water through the coils at 180°F ($\pm 10^\circ$) and 60 (± 20) psi until the internal surfaces indicate a pH within the range of 6 to 8 when contacted by pH indicating paper.

3.6.10 Thoroughly dry the passivated coils by blowing compressed air or nitrogen through the coils until there is no moisture pickup in the pre-filtered drying gas as it passes through the coils.

3.6.11 Heat exchanger assembly - Cleaning and recleaning procedure for interior of tubing are as follows.

3.6.11.1 Thoroughly degrease coils internally by circulating Type I trichloroethylene at ambient temperature through the coils at 60 (± 20) psi for 5 to 10 minutes. Sample the trichloroethylene during the last 2 minutes it is being circulated through the coils. Use respirators unless room exhaust is available as a safety precaution. Use of gloves is mandatory for this operation.

3.6.11.2 Using 500 ml of the trichloroethylene sample obtained in 3.6.11.1, test as specified in paragraphs 4.4.2.1 and 4.4.2.2 of Specification MSFC-SPEC-164 to assure that the heat exchanger coils meet the particle size and NVR requirements of Specification MSFC-SPEC-164. Use gloves and respirators unless room exhaust is available as a safety precaution. If additional recleaning is required then, the tubing must be cleaned in accordance with paragraphs 3.6.11.1, 3.6.11.2 and 3.6.11.3.

3.6.11.3 Position the clean heat exchanger in an upright position, then dry by blowing compressed air or nitrogen through the coils at 60 (± 20) psi and 180°F ($\pm 20^\circ$) for 30 to 60 minutes.

3.6.11.4 Vacuum dry in accordance with Specification MS 101.0

3.6.11.5 Repeat paragraph 3.6.11.3.

3.6.11.6 Seal openings at the LOX inlet flange, GOX outlet flange, and both end openings in the body of the heat exchanger assembly with a minimum of two thicknesses of aluminum foil reinforced with tape. Put sealed heat exchanger assembly into individual cartons or crates for storage until ready for installation.

4. QUALITY ASSURANCE PROVISIONS

4.1 Acceptance Inspections

4.1.1 Visual inspections - The internal surfaces of all cleaned heat exchanger tubing intended for contact with LOX or GOX shall be visually inspected for the presence of moisture and foreign materials such as corrosion, scale, dirt, oil, grease, and similar foreign materials. The necessary special equipment shall be utilized to assure that internal surfaces are actually clean. The presence of visible contamination shall be cause for re-cleaning of the heat exchanger by the LOX cleaning process, excluding the use of the acid pickling solution. Scale-free discoloration due to welding and heat treatment is permitted.

4.1.2 Surface acidity or alkalinity - The internal surfaces of all cleaned heat exchanger tubing shall be tested with pH indicating paper, while the coil is still wet from the last rinse to determine that the surface acidity or alkalinity registers pH between 6 and 8. The pH indicating paper used shall be sensitive to the requirement specified above.

4.2 Reliability Tests

4.2.1 Particle size - Reliability test samples for particle size shall be prepared as specified in 4.4.1 and tested as specified in paragraph 4.4.2.1 of Specification MSFC-SPEC-164.

4.2.2 Nonvolatile residue - Reliability test samples for nonvolatile residue shall be prepared as specified in paragraph 4.4.1 and tested as specified in paragraph 4.4.2.2 of Specification MSFC-SPEC-164.

5. PREPARATION FOR DELIVERY

5.1 Preservation and Packaging - Heat exchangers and heat exchanger components, such as tubes and fittings, shall be packaged immediately upon completion of the cleaning and drying processes in order to prevent contamination. All openings into the exchanger as well as surfaces intended for service in contact with LOX or GOX shall be protected by covering these areas with a minimum of two layers of aluminum foil reinforced with tape. The aluminum-foil-protected heat exchanger assemblies shall then be packed in individual wooden or metal containers capable of providing absolute protection against mechanical damage during storage, handling, or transportation before installation.

5.2 Marking - Cleaned heat exchangers and their components shall be marked with tags conforming to Type C, Specification UU-T-81D(1), and shall contain the following information.

- Part or identification number
- Method of cleaning and micron level
- Date of cleaning
- Title, date, and number of this manufacturing specification
- Contractor identification
- Manufacturer's serial number
- Service medium or intended use of heat exchanger

6. NOTES

6.1 Intended Use - This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use in cleaning and preserving of stainless steel heat exchangers for use with liquid and gaseous oxygen. Absolute cleanliness is essential as any contamination in heat exchanger lines may cause serious malfunctions in the operation of the space launch vehicle.

NOTICE: When government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related government procurement operation, the United States Government incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have

formulated, furnished or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

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MANUFACTURING SPECIFICATION FOR
CLEANING AND PASSIVATION OF AUSTENITIC (300 SERIES)
STAINLESS STEEL SUCTION LINES

1. SCOPE

1.1 Scope - This manufacturing specification covers the approved PE Laboratory requirements for the cleaning and passivation of austenitic (300 series) stainless steel suction lines for use in oxygen, fuel, and pneumatic systems for space launch vehicle applications.

1.2 Applicability - The cleaning and passivation procedures outlined in this manufacturing specification are applicable to all austenitic (300 series) stainless steel alloys, with exception of Type 303, in all forms except castings.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this specification, form a part of this specification.

SPECIFICATIONS.

Federal

O-A-51c	Acetone, Technical
O-A-88 (1)	Acid, Nitric, Technical Grade
O-C-303b	Chromium Trioxide, Technical (Chromic Acid)
O-H-795 (2)	Hydrofluoric Acid, Technical
O-P-94A	Paper, Test, pH Indicator
O-P-313	Phosphoric Acid, Technical Grade
O-S-595A (3)	Sodium Dichromate, Technical Grade

O-T-634A	Trichloroethylene, Technical
BB-N-411A	Nitrogen
QQ-S-763B (1)	Steel Bars, Shapes, and Forgings, Corrosion Resisting
QQ-S-766C (Navy Ships) (1)	Steel Plates, Sheets, and Strip, Corrosion Resisting
QQ-W-423	Wire, Steel, Corrosion Resisting
UU-T-81F (1)	Tags, Shipping and Stock
PPP-T-60 (3)	Tape, Pressure Sensitive Adhesive, Waterproof, for Packaging and Sealing

Military

MIL-A-00148B (ORD)	Aluminum Foil
MIL-S-862B (1)	Steel Bars, Corrosion Resisting, and Steel Billets, Corrosion Resisting, Reforging Application
MIL-S-4043 (1) (USA F)	Steel, Corrosion Resisting, (Extra Low Carbon, Type 304), Plate, Sheet and Strip
MIL-R-5031A (1)	Rods and Wire, Welding, Corrosion and Heat Resistant Alloys
MIL-S-5059A (1) (ASG)	Steel, Corrosion Resistant (18-8) Plate, Sheet and Strip
MIL-T-5695B (ASG)	Tubing, Steel, Corrosion Resistant (304), Hardened
MIL-S-6721A	Steel, Corrosion and Heat Resistant (18-8 Stabilized), Plate, Sheet, and Strip (For Aircraft Applications)

BB-N-411A	Nitrogen
QQ-S-763B(1)	Steel Bars, Shapes, and Forgings (Corrosion Resisting)
UU-T-81D(1)	Tags, Shipping, and Stock
PPP-T-60(2)	Tape, Pressure Sensitive Adhesive, Waterproof, for Packaging and Sealing

Military

MIL-A-00148B(ORD)	Aluminum Foil
MIL-S-6712A	Steel, Corrosion and Heat Resistant (18-8 Stabilized), Plate, Sheet, and Strip (For Aircraft Applications), (321 or 347)
MIL-T-6845 (5)	Tubing, Steel, Corrosion Resisting (18-8) Aircraft, Hydraulic System
MIL-D-16791C	Detergents, Nonionic

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MSFC-SPEC-131	Tubing, Steel, Corrosion-Resistant (304 and 304L) Seamless
MSFC-SPEC-164	Cleanliness of Components for use in Liquid Oxygen, Fuel, and Pneumatic Systems
MS 101.0	Manufacturing Specification for Drying of Components in a Vacuum

MIL-T-6737A (2) (ASG)	Tubing, Steel, Corrosion and Heat Resistant (18-8 Stabilized), Welded
MIL-T-6845 (5)	Tubing, Steel, Corrosion Resisting (18-8) Aircraft Hydraulic System
MIL-S-7720 (1)	Steel, Corrosion Resistant (18-8) Bars and Forgings Stock (For Aircraft Applications)
MIL-T-7880 (2)	Tubing, Flexible, Corrosion Resistant and Heat Resistant Steel
MIL-T-8504 (2) (ASG)	Tubing, Steel, Corrosion Resistant (18-8) Annealed, Aircraft Hydraulic System
MIL-T-8506 (1)	Tubing, Steel, Corrosion Resistant, (304) Annealed
MIL-T-8606A	Tubing, Steel, Corrosion Resistant (18-8 Stabilized)
MIL-T-8808A (ASG)	Tubing, Steel, Corrosion Resistant (18-8 Stabilized), Aircraft Hydraulic Quality.
MIL-D-16791C	Detergents, Nonionic
MIL-F-20138B	Floor Plate, Stainless Steel, Rolled
MIL-T-52010 (ORD)	Tube, Corrosion-resisting, Steel, Seamless and Welded

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10509305	Cleanliness of Components for Use in LOX, Fuel and Pneumatic Systems
MS 101.0	Manufacturing Specification for The Drying of Components in a Vacuum

2.2 Other Publications - The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

Society of Automotive Engineers, Inc.

AMS-5514A	Plate, Sheet and Strip, 18 Cr - 11 Ni Deep Drawing and Spinning (Type 305)
AMS-5521B	Plate, Sheet and Strip, 25 Cr - 20 Ni, Deep Drawing and Spinning, (Type 310)
AMS-5522B	Plate, Sheet and Strip, 25 Cr - 20 Ni - 2 Si (Type 314)
AMS-5523	Plate, Sheet and Strip, 23 Cr - 13.5 Ni, (Type 309S)
AMS-5556A	Tubing, Hydraulic, 18 Cr - 11 Ni (Cb+Ta), (Type 347)
AMS-5557A	Tubing, Hydraulic, 18 Cr - 11 Ni - Ti (Type 321)
AMS-5558	Tubing, Welded, 18 Cr - 11 Ni (Cb+Ta) Thin Wall, (Type 347)
AMS-5559	Tubing, Welded, 18 Cr - 10 Ni - Ti Thin Wall, (Type 321)
AMS-5572B	Tubing, Seamless, 25 Cr - 20 Ni, (Type 310)
AMS-5573B	Tubing, Seamless, 17 Cr - 12.5 Ni, 2.3 Mo, (Type 316)
AMS-5574	Tubing, Seamless, 23 Cr - 13.5 Ni, (Type 309S)

AMS-5577A	Tubing, Welded, 25 Cr - 20 Ni, (Type 310)
AMS-5649	Bars and Forgings, 18 Cr - 13 Ni - 2.1 Mo, Free Machining, (Type 316 FM)
AMS-5650A	Bars, Forgings, and Mechanical Tubing, 23 Cr - 13.5 Ni, (Type 309S)
AMS-5652B	Bars, Forgings, and Mechanical Tub- ing 25 Cr - 20 Ni - 2 Si, (Type 314)
AMS-5686A	Wire, Riveting, Annealed, 18 Cr - 11 Ni, Solution Heat Treated, (Type 305)
AMS-5689	Wire, Screen, 18 Cr - 9.5 Ni - Ti, (Type 321)

(Copies of the specifications listed above may be obtained from the Society of Automotive Engineers, Inc., 485 Lexington Avenue, New York 17, N. Y.)

Turco Products, Inc.

Tech, Product Data Bulletin No. A-76	Turco Vitro-Klene
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(Copies of the publication listed above may be obtained from Turco Products, Inc., P. O. Box 1055, Wilmington, California.

3. REQUIREMENTS

3.1 General - No deviation from the requirements of this manufacturing specification shall be permitted without prior written approval of the MSFC initiating activity.

3.2 Materials

3. 2. 1 Trichloroethylene for vapor degreasing - The trichloroethylene used for vapor degreasing shall meet all of the requirements for Type II of Specification O-T-634a.

3. 2. 2 Trichloroethylene for flushing or immersion degreasing - The trichloroethylene used for flushing or immersion degreasing shall meet all of the requirements for Type I of Specification O-T-634a, except that the non-volatile residue content shall not be greater than 0.010 gram per 500 ml when tested in the manner specified in paragraph 4. 4. 2. 2 of Specification 10509305. In addition, no 500 ml sample shall contain a particle larger than 175 microns in any dimension or more than 5 particles between 100 and 175 microns in any dimension.

3. 2. 3 Nonionic detergent - The nonionic detergent used for cleaning shall meet all of the requirements for Type I of Specification MIL-D-16791C. (The only detergent approved for use at this time is Dowell F-33.)

3. 2. 4 Demineralized water - The minimum specific resistance of demineralized water shall be 50000 ohm. It shall contain no particles larger than 175 microns in any dimension.

3. 2. 5 Drying or preservation gases - Compressed gases used in drying or preserving of stainless steel shall contain no particles larger than 100 microns (absolute), the oil content of the gases shall not be greater than 3 parts per million (by weight), and the moisture content of the gases shall not be greater than 24 parts per million (by volume).

3. 2. 5. 1 Nitrogen - Nitrogen gas, to be used in drying or preservation processes, shall conform to Type I, Class B of Specification BB-N-411a, except that the particle size, oil content, and moisture content requirements of paragraph 3. 2. 5 shall be met.

3. 2. 5. 2 Air - Compressed air, used for drying or preservation of stainless steel, shall conform to the requirements of paragraph 3. 2. 5 above.

3. 2. 6 Paper, pH indicating - The pH indicating paper used in determining the acidity or alkalinity of stainless steel surfaces shall be of a grade conforming to range a, b, c, e, f, or short range of Specification O-P-94a.

3. 2. 7 Aluminum foil - The aluminum foil used in the protection of cleaned stainless steel shall be of the dry annealed type conforming to Specification MIL-A-00148B (ORD).

3.2.8 Tape - The tape, used to secure and reinforce protective coverings on cleaned stainless steel, shall conform to Type I of Specification PPP-T-60 (3).

3.2.9 Tags - The tags, used to mark and identify cleaned stainless steel, shall be Type B, Class I of Specification UU-T-81F (1).

3.3 Surface Alkalinity or Acidity - Austenitic stainless steel material or components, which have been subjected to either an alkaline or an acid cleaning solution, shall be rinsed with demineralized water until all surfaces of the material or component register a pH within the range of 6 to 8 when contacted by pH indicating paper.

3.4 Level of Cleanliness and Dryness - Austenitic stainless steel cleaned and passivated by the procedures outlined in this manufacturing specification shall meet all of the particle size, and nonvolatile residue requirements of Specification 10509305.

3.5 Cleaning and Passivation After Butt Welding

3.5.1 Material or parts not combined with gimbals, inner liners, braided hose, flexible hose, teflon-lined hose or brazed joints.

3.5.1.1 Using a stainless steel wire brush or stainless steel wool free of iron particles, remove all loose weld scale from external welds only, and the areas immediately adjacent to all welds. Heat-tint due to welding need not be removed.

3.5.1.2 Vapor degrease the fusion welded part or assembly in the manner outlined in Specification MS-150.0.

3.5.1.3 Air dry the thoroughly degreased part or assembly by allowing it to hang above the vapor degreasing tank until all liquid evaporates.

3.5.1.4 Immerse the thoroughly dried part or assembly in an alkaline cleaning solution (Turco Vitro-Klene, or equal) containing 8 to 10 ounces of cleaner per gallon of water at 200 to 212° F for 10 to 15 minutes.

3.5.1.5 Rinse the cleaned part or assembly by spraying or by immersing in tap water at 180 °F (±20°) for 3 to 5 minutes.

3.5.1.6 Pickle the rinsed part or assembly by immersing in an aqueous solution containing 20 (± 5) percent by volume of nitric acid (sp. gr. 1.42) and 1.5 (± 0.5) percent by volume of hydrofluoric acid (60 percent strength) at a temperature of 90°F ($\pm 15^\circ$) for 30 to 60 minutes. Do not pickle parts possessing a smooth and rust free finish.

3.5.1.7 Rinse the pickled part or assembly by spraying or by immersing in ambient temperature tap water for 15 to 20 minutes.

3.5.1.8 Passivate the rinsed part or assembly by immersing in an aqueous solution containing 20 (± 5) percent by volume of nitric acid (sp. gr. 1.42) and 2 (± 0.5) percent by weight of sodium dichromate at 140°F ($\pm 5^\circ$) for 30 to 60 minutes.

3.5.1.9 Rinse each passivated part or assembly by immersing in demineralized water at ambient temperature for 5 to 10 minutes. After immersion, spray rinse with demineralized water until the part or assembly surfaces register a pH within the range of 6 to 8 when contacted by pH indicating paper. This rinsing step, followed by drying, as specified in 3.5.1.12 below, completes the cleaning and passivation requirements of stainless steel.

3.5.1.10 After thorough rinsing, 500 ml samples of demineralized water shall be flushed over each square foot of surface area which is to be in contact with fuel or pneumatic environments. At least one of these 500 ml samples shall be checked for particle size in the manner specified in paragraph 4.4.2.1 of Specification 10509305. Particles of contamination per square foot of area cleaned and tested shall not exceed the following limits.

- No particle larger than 2500 microns in any dimension
- One particle between 700 and 2500 microns in its largest dimension
- Five particles between 175 and 700 microns in their largest dimension

3.5.1.11 If the particle size determination of 3.5.1.10 indicates that additional cleaning is required, repeat the rinsing steps of 3.5.1.9 until additional samples do meet the particle size requirements of 3.5.1.10.

3.5.1.12 Thoroughly dry the rinsed stainless steel part or assembly by blowing with air or nitrogen at 180°F ($\pm 20^\circ$) until no moisture can be visually detected on the surface. Redry in a vacuum oven in the manner outlined in Specification MS 101.0. This drying step followed by packaging, as specified in 3.5.1.13, completes the cleaning and passivation requirements for stainless steel to be used in fuel or pneumatic applications. Stainless steel for use in oxygen systems shall receive additional cleaning as specified in 3.5.3 below.

3.5.1.13 Protect cleaned fuel and pneumatic system stainless steel components from contamination by covering all openings and contact surfaces with a minimum of two layers of aluminum foil secured and reinforced with tape. Small parts or assemblies of stainless steel shall then be placed in clean polyethylene bags, the bags purged with nitrogen or air, the end of the bag heat sealed to assure an inert package during storage, and packed as necessary to prevent damage during storage or handling.

3.5.1.14 Stainless steel material or components intended for service in contact with liquid or gaseous oxygen shall be cleaned as specified in paragraph 3.5.3.

3.5.2 Material or parts combined with gimbals, inner liners, braided hose, flexible hose, Teflon-lined hose or brazed joints

3.5.2.1 Using a stainless steel wire brush or stainless steel wool free of iron particles, remove all loose weld scale from external welds only, and from the areas immediately adjacent to all welds. Heat tint due to welding need not be removed.

3.5.2.2 Vapor degrease the fusion welded part or assembly in the manner outlined in Specification MS-150.0.

3.5.2.3 Air dry the thoroughly degreased part or assembly by allowing it to hang above the vapor degreasing tank until all liquid evaporates.

3.5.2.4 Immerse the thoroughly dried parts or assembly in an alkaline cleaning solution (Turco Vitro-Klene, or equal) containing 8 to 10 ounces of cleaner per gallon of water at 200 to 212°F for 10 to 15 minutes.

3.5.2.5 Rinse the cleaned part or assembly by spraying or by immersing in tap water at 180°F ($\pm 20^\circ$) for 3 to 5 minutes.

3. 5. 2. 6 Rinse each part or assembly and check the surface pH as specified in paragraph 3. 5. 1. 9. This step followed by drying, as specified in paragraph 3. 5. 2. 8 below, completes the cleaning of parts or assemblies.

3. 5. 2. 7 For parts or assemblies to be used in fuel or pneumatic applications, check for particle contamination as specified in paragraph 3. 5. 1. 10 and 3. 5. 1. 11.

3. 5. 2. 8 Dry all parts or components as specified in paragraph 3. 5. 1. 12. Stainless steel, for use in oxygen systems, shall receive additional cleaning as specified in 3. 5. 3 below.

3. 5. 2. 9 Protect and package all fuel and pneumatic parts or components as specified in paragraph 3. 5. 1. 13:

3. 5. 2. 10 Clean and package all oxygen system parts or components as specified in paragraph 3. 5. 3

3. 5. 3 LOX-service cleaning

3. 5. 3. 1 Clean by vapor degreasing in the manner outlined in Specification MS 150. 0.

3. 5. 3. 2 Thoroughly rinse the degreased stainless steel by flushing with new Type I trichloroethylene at ambient temperature for 3 to 5 minutes.

3. 5. 3. 3 After thorough flushing, a 500 ml sample of new Type I trichloroethylene shall be flushed through or over the surfaces which will later be in contact with liquid or gaseous oxygen environments.

A. This 500 ml sample shall be checked for particle size in the manner specified in paragraph 4. 4. 2. 1 of Specification 10509305. Particle contamination per square foot of surface area tested shall be as specified in 3. 5. 1. 10.

B. After filtering for particle size determination, the 500 ml sample shall be checked for nonvolatile residue pickup in the manner specified in paragraph 4. 4. 2. 2 of Specification 10509305. The nonvolatile residue contamination on the surfaces intended for contact with liquid or gaseous oxygen shall not be greater than 0. 001 gram per square foot.

3.5.3.4 Thoroughly dry the degreased stainless steel by blowing with air or nitrogen at 180° F ($\pm 20^\circ$) until no moisture can be visually detected on the surface.

3.5.3.5 Vacuum dry the air-dried stainless steel material, part or assembly in a vacuum oven in the manner outlined in Specification MS 101.0.

3.5.3.6 Seal all openings and protect all exposed surfaces with a minimum of two thicknesses of aluminum foil secured and reinforced with tape. Pieces of material, parts, and assemblies which are small enough shall be placed in clean polyethylene bags, the bags purged with nitrogen or air, the end of the bag heat sealed to assure an inert package during storage, and overpacked as necessary to prevent damage during storage or handling.

3.6 Cleaning Before Annealing or Stress Relieving (Parts Not Combined with Gimbals, Inner Liners, Braided Hose, or Flexible Joints)

3.6.1 Scale removal - Using a stainless steel wire brush or stainless steel wool free of iron particles, remove all loose weld scale from the surfaces of the stainless steel (external welds only as part of tubing).

3.6.2 Preliminary degreasing - Using a clean cloth or new stainless steel wool saturated with unused or redistilled acetone, remove all inked lettering, wax crayon or pencil markings, spots of grease, heavy oils, and forming lubricants such as Lubri-Plate or wax from all surfaces of the stainless steel.

3.6.3 Vapor degrease - Vapor degrease the partially cleaned stainless steel in the manner outlined in Specification MS 150.0

3.6.4 Dry - Dry the thoroughly degreased material by allowing it to evaporate to dryness while hanging above the degreasing tank.

3.6.5 Alkaline clean - Immerse the stainless steel in an alkaline cleaning solution (Turco Vitro-Klene, or equal) containing 8 to 10 ounces of cleaner per gallon of water at 200 to 212 F for 10 to 15 minutes.

3.6.6 Rinse - Rinse by spraying or by immersing in tap water at 180°F ($\pm 20^\circ$) for 3 to 5 minutes.

3.6.7 Dry - Air dry the cleaned and rinsed stainless steel by allowing it to evaporate to dryness while hanging above the rinse tank.

3.6.8 Protect - Protect the dried stainless steel material, part, or component from recontamination by covering all openings or exposed surfaces with a minimum of two thicknesses of aluminum foil secured and reinforced with tape. Deliver the protected stainless steel material, part, or component promptly to the Heat Treat Department for heat treatment.

3.7 Cleaning and Passivation After Annealing or Stress Relieving (Parts not combined with Gimbals, Inner Liners, Braided Hose, or Flexible Joints)

3.7.1 Preliminary degreasing - Examine heat treated stainless steel for signs of fresh oil or grease contamination after heat treatment. If no fresh contamination can be found, proceed to paragraph 3.7.4. If fresh contamination is found, degrease as specified in paragraph 3.7.2.

3.7.2 Degreasing - Vapor degrease the oil or grease contaminated material, part, or component in the manner outlined in Specification MS 150.0.

3.7.3 Dry - Air dry the thoroughly degreased stainless steel by allowing it to evaporate to dryness.

3.7.4 Pickle - Pickle to remove scale by immersing in an aqueous solution containing 20 (± 5) percent by volume of nitric acid (sp. gr. 1.42) and 1.5 (± 0.5) percent by volume of hydrofluoric acid (60 percent strength) at a temperature of 90°F ($\pm 15^\circ$) for 30 to 60 minutes. Scrub part while immersed as necessary to remove oxide or scale.

3.7.5 Rinse - Rinse the pickled stainless steel by spraying or by immersing in tap water at 180°F ($\pm 20^\circ$) for 10 to 15 minutes.

3.7.6 Passivate - Passivate by immersing for 30 minutes in a solution containing 20 percent by volume of nitric acid (sp. gr. 1.42) and 2 percent by weight of sodium dichromate at 125°F ($\pm 5^\circ$).

3.7.7 Rinse - Rinse by spraying or immersing in 180°F ($\pm 20^\circ$) tap water.

3.7.8 Passivate - Passivate by immersing for 1 hour in an aqueous solution containing 5 percent by weight of sodium dichromate at 150°F ($\pm 10^\circ$).

3.7.9 Rinse - Rinse by spraying or by immersing in demineralized water at ambient temperature.

3.7.10 Seal - Seal by immersing in a 180° F (±20) rinse maintained at a pH within the range of 3 to 5 by the addition of flake chromic acid.

3.7.11 Dry - Air dry the passivated part or assembly by allowing it to hang above the rinse tank until all liquid evaporates.

3.7.12 Particle determination - Samples for the determination of particle contamination shall be taken, tested, and shall meet the requirements specified in 3.5.1.10 for all material intended for service in contact with fuel or pneumatic environments.

3.7.13 Additional cleaning - If the particle size determination of 3.7.12 indicates additional cleaning is required, repeat the rinsing and drying of paragraphs 3.7.9 and 3.7.11 until additional samples do meet the particle size requirements of paragraph 3.7.12.

3.7.14 Dry - Thoroughly dry rinsed stainless steel in the manner specified in paragraph 3.5.1.12. This drying operation, followed by packaging as specified in paragraph 3.7.15 below, completes the cleaning and passivation requirements for heat treated stainless steel to be used in fuel or pneumatic applications. Heat-treated stainless steel, for use in oxygen systems, shall receive additional cleaning as specified in paragraph 3.7.16.

3.7.15 Protect - Protect and package cleaned, heat-treated fuel, and pneumatic system stainless steel components as specified in paragraph 3.5.1.13.

3.7.16 LOX-service cleaning - Heat-treated stainless steel, for use in oxygen systems, shall be degreased, rinsed, checked for particle size, checked for nonvolatile residue, dried, protected, and packaged as specified in paragraph 3.5.3.

3.8 Cleaning and Passivation of Tubing 2 Inches or Less in External Diameter (Parts Not combined With Inner Liners, Braided Hose, Flexible Joints, or Gimbals)

3.8.1 Examination - Visually examine interior of tubing for the presence of weld scale or oxides due to previous fabrication operations.

3.8.2 Scale removal - If welding or heat-treating scale (not heat tint) is found to be present, mechanically remove by buffing with a 300-series stainless steel wire brush free of iron particles (external welds only for tubing).

3.8.3 Degrease - Degrease long sections of tubing by circulating Type I trichloroethylene through the tubing at ambient temperature and 15 to 20 psi pressure for 5 to 10 minutes. Degrease short tubing sections by vapor degreasing in the manner outlined in Specification MS 150.0.

3.8.4 Dry - Air dry the degreased tubing sections by allowing them to evaporate to dryness. Dry long sections of tubing by blowing air at 180°F ($\pm 20^\circ$) through the tubing for 20 to 30 minutes.

3.8.5 Examine - Visually examine each section of tubing for the presence of aluminum flanges, which must be protected from the pickling and passivating solutions, used in cleaning and passivating stainless steel lines.

3.8.6 Masking - If flanges are found in 3.8.5 which require protection from pickling and passivating solutions, mask all aluminum flanges only by quickly dipping into a bath of cellulose acetate (Quick-dip, or equal) at 300 to 350°F followed by air drying.

3.8.7 Alkaline cleaning - Chemically clean long sections of tubing by circulating an alkaline cleaning solution (Turco Vitro-Klene, or equal) containing 8 to 10 ounces of cleaner per gallon of water at 200 to 212°F for 10 to 15 minutes and at a pressure of 60 (± 20) psi. Short tube sections shall be immersed in Turco Vitro-Klene, or equal, containing 8 to 10 ounces of cleaner per gallon of water at 200 to 212°F for 10 to 15 minutes.

3.8.8 Rinse - Rinse chemically cleaned tubing by circulating tap water through, or immersing the tubing in tap water, at 180°F ($\pm 20^\circ$) for 3 to 5 minutes.

3.8.9 Pickle - Pickle tubing by circulating an aqueous solution containing 20 (± 5) percent (by volume) of nitric acid (sp. gr. 1.42) and 1.5 (± 0.5) percent (by volume) of hydrofluoric acid (60 percent strength) through the tubing at a pressure of 60 (± 20) psi and at a temperature of 90°F ($\pm 15^\circ$) for 10 (± 2) minutes. Short tube sections shall be immersed in the pickling solution instead of circulating the solution through its interior.

3.8.10 Rinse - Rinse by circulating tap water through or by immersing in tap water at 180°F ($\pm 20^\circ$) for 15 to 20 minutes.

3. 8. 11 Passivate - Passivate tubing by circulating an aqueous solution containing 20 (± 5) percent (by volume) of nitric acid (sp. gr. 1.42) and 2 (± 0.5) percent (by weight) of sodium dichromate through the tubing at 60 (± 20) psi and 140° F ($\pm 5^\circ$) for 30 to 60 minutes. Short lengths of tubing shall be immersed in the passivating solution instead of circulating the solution through the tubing.

3. 8. 12 Rinse - Thoroughly rinse long lengths of tubing by circulating demineralized water through the tubing until all internal surfaces of the tubing register a pH within the range of 6 to 8 when contacted by pH indicating paper. Rinse short lengths of tubing by immersing and spraying with demineralized water until all surfaces register a pH within the range of 6 to 8 when contacted by pH indicating paper.

3. 8. 13 Remove maskant - If cellulose acetate maskant has been used on any part of the tubing, strip the maskant from the masked areas and rinse the entire tubing section with demineralized water for an additional 3 to 5 minutes. This rinse, followed by drying as specified in paragraph 3. 8. 16 below, completes the cleaning and passivation required for tubing.

3. 8. 14 Particle size checking - After thorough rinsing, samples for the determination of particle contamination shall be taken, tested, and shall meet the requirements specified in paragraph 3. 5. 1. 10 for all tubing intended for service in contact with fuel or pneumatic environments.

3. 8. 15 Additional cleaning - If the particle size determination of 3. 8. 14 indicates that additional cleaning is required, repeat the rinses of 3. 8. 12 and 3. 8. 13 until additional samples do meet the particle size requirements of paragraph 3. 8. 14.

3. 8. 16 Dry - Thoroughly dry the rinsed and tested tubing in the manner specified in 3. 5. 1. 12. This drying operation, followed by packaging as specified in 3. 8. 17 below, completes the cleaning and passivation requirements for tubing to be used in fuel or pneumatic applications. Tubing for use in oxygen systems shall receive additional cleaning as specified in paragraph 3. 8. 18 through 3. 8. 25.

3. 8. 17 Protect - Protect and package short sections of cleaned fuel and pneumatic system tubing as specified in paragraph 3. 5. 1. 13. Lengths of tubing too long to be sealed in polyethylene bags shall have both ends sealed by a minimum of two thicknesses of aluminum foil reinforced and secured by tape.

3. 8. 18 Detergent cleaning - Circulate an aqueous solution containing 0. 5 to 1. 0 percent by volume of nonionic detergent through the interior of the tubing at 180° F ($\pm 20^\circ$) for 10 to 20 minutes.

3. 8. 19 Rinse - After thorough detergent cleaning, rinse thoroughly by circulating demineralized water through the interior of the tubing at ambient temperature for 10 to 20 minutes.

3. 8. 20 Dry - Dry the thoroughly rinsed tubing in the manner specified in paragraph 3. 5. 3. 4.

3. 8. 21 Degrease - Circulate Type I trichloroethylene through the interior of the tubing at ambient temperature and 15 to 20 psi for 20 to 30 minutes. Sample the trichloroethylene from the effluent side of the circulating system for NVR test samples.

3. 8. 22 Tests - The sample taken from tubing intended for use in oxygen systems shall be checked for particle size, checked for nonvolatile residue, and the tubing dried, protected, and packaged as specified in paragraphs 3. 5. 3. 5 and 3. 5. 3. 6.

3. 9 Cleaning of Tubing and Components Over 2 inches in External Diameter

3. 9. 1 Visual examination - Visually examine the interior of tubing or components for the presence of weld oxide, scale, or heat tint due to previous fabrication or forming operations.

3. 9. 2 Scale removal - If welding or heat treating scale (not heat tint) is found to be present, mechanically remove by buffing with a 300 series stainless steel wire brush free of iron particles (external welds only for tubing).

3. 9. 3 Positioning - Place long sections of tubing or components in vertical position then flange the openings with funnel-type, open flanges. Insert a Teflon-lined flexible hose and nozzle through the opening in the flange at the top of the vertical length of tubing or component. The nozzle and attached flexible hose and fittings shall have a protective covering of Teflon and be surrounded by Teflon bumpers.

3. 9. 4 Detergent cleaning - Clean the interior of the tubing or component by spraying a 0. 1 percent detergent solution through the nozzle at 200 (± 50)

psi pump pressure at ambient temperature for a minimum of 30 minutes. Lower and raise the nozzle through at least three top-to-bottom cycles while detergent solution is being sprayed.

3. 9. 5 Rinse - After thorough detergent cleaning, spray rinse the interior of the tubing or component by spraying demineralized water through the nozzle at 200 (\pm 50) psi and ambient temperature for a minimum of 30 minutes. Lower and raise the nozzle through at least three top-to-bottom-to-top cycles while the demineralized water is being sprayed. This rinsing, followed by drying as specified in 3. 9. 9 below, completes the cleaning of components and tubing over 2 inches in external diameter.

3. 9. 6 Sampling - During the last 5 minutes of the demineralized water rinse cycle, the discharge water from the tubing or component shall be continuously sampled to obtain samples for particle size determination.

3. 9. 7 Particle contamination - The rinse samples for the determination of particle size contamination shall be tested and shall meet the requirements specified in 3. 5. 1. 10 for all components and tubing intended for service in contact with fuel or pneumatic environments.

3. 9. 8 Additional cleaning - If the particle size determination of 3. 9. 7 indicates that additional cleaning is required, repeat steps 3. 9. 4 through 3. 9. 6 until additional samples do meet the particle size requirements of paragraph 3. 9. 7.

3. 9. 9 Dry - Thoroughly dry the rinsed and tested component or tubing in the manner specified in 3. 5. 1. 12. This drying operation, followed by protecting and packaging as specified in 3. 9. 10 below, completes the cleaning requirements for components and tubing over 2 inches in external diameter to be used in fuel or pneumatic applications. Components or tubing for use in oxygen systems shall receive additional cleaning as specified in paragraph 3. 9. 11 through 3. 9. 14.

3. 9. 10 Protect - Protect and package sections of cleaned fuel and pneumatic system components and tubing as specified in paragraphs 3. 5. 1. 13 and 3. 8. 17.

3. 9. 11 LOX-service cleaning - Lengths of tubing or components for use in oxygen systems shall be degreased by sealing both ends of the component or tubing with LOX-clean Teflon gaskets and flanges, then circulating or

spraying Type I trichloroethylene through the component or tubing at ambient temperature to contact all internal surfaces for a minimum of 30 minutes. Hoses leading into and out of the flanges must be LOX-clean before each cleaning operation.

3.9.11.1 Protect all bellows or expansion joints, which are a part of the component or tubing, by wrapping with felt.

3.9.12 NVR checking - After thoroughly circulating or spraying Type I trichloroethylene through the component or tubing, sample trichloroethylene from the effluent side for particle and NVR determinations.

3.9.13 Checking, drying, and protecting - The trichloroethylene sample taken shall be checked for non-volatile residue pickup and the component or tubing dried, protected, and packaged as specified in paragraphs 3.5.3.3.2, 3.5.3.4, 3.5.3.5, and 3.5.3.6.

3.10 Cleaning, Pickling, and Passivating of Specific Areas of Stainless Steel Assemblies, Excluding Lap-Weld, Gimbal, Inner-Liner and Flex Joint Areas

3.10.1 Vapor degrease - Vapor degrease the assembly in the manner outlined in Specification MS 150.0.

3.10.2 Scale removal - Inspect for and remove welding or heat treating oxide or scale with a stainless steel wire brush free of iron particles (external welds only for tubing).

3.10.3 Plugging - Place a specially constructed pneumatic plug (O.D. of plug smaller than I.D. of assembly) inside the tubing or component and at least 2 inches below area to be cleaned, pickled, and passivated. Secure the plug by inflating with compressed air to a maximum pressure of 100 psi.

3.10.4 Leak checking - Fill the assembly above the plug with water, or spray the plug with water, and inspect for leaks around edges of the plug. Pour or siphon the water from above the plug when sure that no leaks are present.

3.10.5 Pickle - Pickle the assembly above the plug by filling with an aqueous solution containing 20 (± 5) percent (by volume) of nitric acid (sp. gr. 1.42) and 1.5 (± 0.5) percent (by volume) of hydrofluoric acid (60 percent

strength) at 90° F ($\pm 15^\circ$) for 30 to 60 minutes. Remove the pickling solution by eduction through a Teflon hose.

3.10.6 Neutralize - Neutralize the pickled assembly above the plug with an aqueous solution containing 2 to 3 ounces per gallon of Keylite, or equal. Retain this solution in the pickled assembly at ambient temperature for a minimum of 10 minutes. Remove the Keylite solution by eduction through a Teflon hose.

3.10.7 Rinse - Rinse the pickled assembly above the plug by filling with tap water. Drain this water by eduction. Continue to fill and drain with water until the pickled and rinsed surfaces register a pH within the range of 6 to 8 when contacted by pH indicating paper.

3.10.8 Passivate - Passivate the rinsed assembly above the plug by filling with an aqueous solution containing 20 (± 5) percent by volume of nitric acid (sp. gr. 1.42) and 2 (± 0.5) percent by weight of sodium dichromate of 140° F ($\pm 5^\circ$) for 30 to 60 minutes.

3.10.9 Neutralize - Neutralize the passivated assembly above the plug by filling with a demineralized water solution containing 2 to 3 ounces of Keylite per gallon. Retain this solution in the passivated assembly at ambient temperature for a minimum of 10 minutes. Remove the Keylite solution by eduction through a Teflon hose.

3.10.10 Rinse - Rinse the passivated assembly above the plug by filling with demineralized water. Drain this water by eduction. Continue to fill and drain with demineralized water until the passivated and rinsed surfaces register a pH within the range of 6 to 8 when contacted by pH indicating paper.

3.10.11 Dry - Dry the rinsed assembly above the plug by blowing with 180° F ($\pm 20^\circ$) air or nitrogen until there is no moisture pickup in the drying gas after passing through or over the assembly. Redry in a vacuum oven in the manner outlined in Specification MS 101.0.

3.10.12 Remove plug - Release air pressure, deflate the specially constructed pneumatic plug and remove from the assembly.

3.10.13 LOX-service cleaning - LOX clean the parts in accordance with paragraphs 3.9.11, 3.9.12, and 3.9.13.

4. MANUFACTURING CONTROL PROVISIONS

4.1 Acceptance Inspections

4.1.1 Visual examination - All cleaned stainless steel components shall be visually inspected for the presence of moisture and foreign materials such as corrosion, scale, dirt, oil, grease, and similar foreign materials. The presence of visible contamination shall be cause for rejection and will necessitate recleaning of the component. Scale free discoloration due to welding or heat treatment is permitted.

4.1.2 Surface acidity or alkalinity - Surfaces of cleaned and passivated components shall be tested with pH indicating paper while the components are still wet from the last rinse or after wetting the test surface with a few drops of distilled water. Surfaces shall register a pH between 6 and 8.

4.2 Reliability Tests

4.2.1 Particle size - Reliability test samples for particle size shall be taken from stainless steel components in the manner specified in paragraph 4.4.1 of Specification 10509305 and tested in the manner specified in paragraph 4.4.2.1 of Specification 10509305.

4.2.2 Nonvolatile residue - Reliability test samples for nonvolatile residue determination shall be prepared in the manner specified in paragraph 4.4.1 of Specification 10509305 and tested in the manner specified in paragraph 4.4.2.2 of Specification 10509305.

5. PREPARATION FOR DELIVERY

5.1 Preservation and Packaging - Stainless steel components for use in space launch vehicles shall be preserved and packaged as specified immediately upon completion of the drying process in order to prevent contamination. All openings and surfaces to be exposed to oxygen, fuel or pneumatic environments shall be protected by covering these areas with a minimum of two thicknesses of aluminum foil, saran wrap, or plastic bags secured and reinforced with tape. The protected components shall be packed into containers capable of providing protection against physical damage during necessary storage, handling or transportation before installation.

5.2 Marking - Cleaned and packaged stainless steel components shall be marked with tags, conforming to Type B of Specification UU-T-81F (1), which shall contain the following information.

- Part or identification number
- Method of cleaning and micron level
- Date of cleaning
- Title, date and number of this manufacturing specification
- Contractor identification
- Manufacturer's serial number
- Service medium or intended use of component

6. NOTES

6.1 Intended Use - This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use in the cleaning of stainless steel suction lines to be used in the fabrication of space launch vehicle components and for the cleaning, passivation, and preservation of these components for use in oxygen, pneumatic, and fuel environments. Absolute cleanliness is essential since any contamination in completed components may result in serious malfunctions in the operation of the space launch vehicle.

NOTICE: When government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related government procurement operation, the United States Government incurs no responsibility nor any obligation; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING SPECIFICATION FOR
ELECTROPOLISHING OF CORROSION RESISTANT STEEL

1. SCOPE

1.1 Scope

This specification covers the approved PE Laboratory manufacturing requirements for electropolishing corrosion resistant steel.

1.2 Applicability

The requirements set forth in this specification are applicable when electropolishing strain hardenable A.I.S.I. 300 series and 19-9DL stainless steel, precipitation hardenable 17-7PH stainless steel, stainless "W" type corrosion resistant steel and A-286 alloy, and A.I.S.I. 400 series.

2. APPLICABLE DOCUMENTS

2.1 Governmental

The following documents, of the issue in effect on the date of use of this specification, form a part of this specification.

SPECIFICATIONS

Federal

O-S-809A	Sulfuric Acid, Technical
O-T-634A	Trichloroethylene, Technical
P-C-535(1)	Cleaning Compound, Plater's Electrocleaning for Steel
QQ-A-677	Anode, Nickel

Military

JAN-A-183(2)	Nitric Acid
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STANDARDS

Federal

151A Metals, Test Methods

Military

MS 36043 Hydrochloric Acid

George C. Marshall Space Flight Center

MS 150.0 Manufacturing Specification for Vapor
Degreasing Metallic Surfaces.

MS 150.1 Manufacturing Specifications for Sand
and Vapor Blasting.

2.2 Other Publications

The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

American Society for Testing and Materials

B254-53 Recommended Practice for Preparation
of and Electroplating on Stainless Steel.

(Copies of this publication may be obtained from the American Society for Testing and Materials, 1916 Race Street, Philadelphia 3, Pa.)

Turco Products, Inc.

A-148 Alkaline Rust Remover

(Copies of this publication can be obtained from Turco Products, Inc., P. O. Box 1055, Wilmington, California.)

Wyandotte Chemicals Corporation

Bulletin F-2780 Wyandotte B. N.

(Copies of this publication can be obtained from Wyandotte Chemicals Corporation, Wyandotte, Michigan.)

3. REQUIREMENTS

3.1 General

No deviation from the requirements of this manufacturing specification shall be permitted without prior written approval of the MSFC initiating activity.

3.2 Materials

3.2.1 Demineralized water

Filtered water, possessing a minimum electrical resistivity of 50 000 ohms, shall be used for final rinses.

3.2.2 Degreasing solvent

Trichloroethylene used for removing mineral or paraffin oils and greases shall meet Specification O-T-634A.

3.2.3 Hot alkaline degreaser

The compound used in an aqueous solution to remove organic soils shall be equal or equivalent to Wyandotte B. N., when the concentration is 4 to 10 ounces per gallon and the temperature is 190° to 210° F. The time of immersion must be the same when a comparison in cleaning efficiency is made.

3.2.4 Electrocleaner

The compound used is an aqueous solution to electroclean parts shall be equal or equivalent to Wyandotte B. N., when the concentration is 4 to 6 ounces per gallon; the temperature, 140° to 160° F; the current density, 20 to 50 amperes per square foot; and the work, cathodic. The period of electrocleaning when making comparisons shall be the same. The cleaning compound must also meet requirements of Specification P-C-535(1).

NOTE: Turco's Alkaline Rust Remover or equivalent when used at a concentration of 8 ounces to 3 pounds per gallon of water at a temperature of 190° F ($\pm 10^\circ$) may be used in lieu of an electrocleaner.

3.2.5 Electropolishing solution

The solution employed for anodically polishing stainless steels shall produce results equal or equivalent to the results obtained from the following reference conditions. A bath composed of 55 percent (by weight) glycolic acid, 30 percent (by weight) sulphuric acid and 15 percent (by weight) water. The specific gravity, at the operating temperature of 170° to 180° F shall be 1.45 (45° Baume'). A current density of 1.0 to 1.25 amperes per square inch being used.

3.2.6 Glycolic acid

The glycolic acid shall be purified glycolic acid.

3.2.7 Sulphuric acid

The sulphuric acid employed shall meet Specification O-S-809A, Type I.

3.2.8 Hydrochloric acid

The hydrochloric acid used shall meet Standard MS 36043.

3.3 Equipment

3.3.1 General

The equipment used in the electropolishing of stainless steels shall permit the attainment of all quality requirements of this specification.

3.3.2 Electrocleaning tank

The tank used for electrocleaning may be constructed of S. A. E. 1010 plate steel with joints welded from both sides.

3.3.3 Rinse tanks

Rinse tanks utilizing either a spray rinse or an overflowing feature may be constructed of S. A. E. 1010 plate steel with joints welding from both sides. However, in the case of the rinse tank following the electropolishing operation, the steel plate shall be protected by rubber, koroseal, or be lined with lead or plastisol.

3.3.4 Electropolishing tank

This tank may be lead lined if constructed of welded S. A. E. 1010 plate steel. It should also be coated with an acid resistant material on its exterior. This construction applies only to the bath given in paragraph 3.2.5. In other cases the tank must be constructed of acid-brick backed up by a lead liner. The tank capacity should be adjusted to a current density of 4 amperes per gallon of solution maximum to avoid overheating.

3.3.5 Preplating activating tank

The tank containing the acid solution for depassivation shall be constructed to type 316L stainless steel and welded with type 321 stainless welding rod.

3.3.6 Anode racks

These may be constructed of lead coated steel, the same type of material being electropolished or steel coated with plastisol or chlorinated polyvinyl chloride. Copper is usable only when chromic acid is absent from the bath and, if the bath is a phosphoric-sulfuric type, the anodes must not be allowed to remain in the solution when they are not in use. Racks shall hold the work so as to avoid gas pockets.

3.3.6.1 Contacts

Work holding clips of phosphorbronze, titanium or tantalum-tungsten alloy are satisfactory, when an ample area of contact is utilized to avoid overheating.

3.3.6.2 Screens

When screens are employed around the work they shall be either constructed of lead or of the same type of material as being polished.

3.3.7 Cathodes:

Cathodes may be constructed of lead strips bolted to the negative electrode or of type 318 stainless steel. In the case of complicated parts, shaped electrodes should be used.

3.3.7.1 Cathode, ceramic diaphragms

Fiberglas mesh is acceptable for a perforated diaphragm around the lead or stainless steel cathodes.

3.3.8 Power supply

A motor-generator set or a selenium rectifier capable of generating a direct current of 2 amperes per square inch of surface polished at 6 to 25 volts is satisfactory.

3.3.9 Agitation

Air agitation may be sufficient in some cases to prevent gas streaks. When mechanical agitation must be used, it may possess a stroke of 4 to 6 inches, a maximum velocity of 50 feet per minute in a horizontal direction and a frequency of 25 cycles a minute.

3.3.10 Ventilation

The air movement over the electropolishing bath shall be identical with that employed over chrome plating tanks. The ventilation over other tanks must be consistent with satisfactory human working environment. When localized, overheating occurs in the electropolishing bath containing glycolic acid, formaldehyde may form and its pungency necessitates effective ventilation.

3.4 Procedure

3.4.1 General

Since electropolishing is accomplished by a procedure that is the opposite of electroplating, and since the work is positive and the dissolved metal is deposited on the cathode, carried in solution or deposited as a precipitate, the following precautions must be observed.

3.4.1.1 Stainless steel types 420, 440A, 440B, and 440C

These shall be stress-annealed at 300° to 700° F before polishing to avoid pickling cracks.

3.4.1.2 Metal lubricants

Copper, lead, and cadmium applied in cold-heating, wire drawing, and spring forming shall be removed by immersion in 20 percent (by volume) nitric acid at 120° to 140° F until the part has gassed for 1 minute.

3.4.1.3 Lubricants

Oil, greases, and fabricating lubricants must be removed per paragraph 3.4.2.2 of this specification, before heat treatment, if heat treatment is performed.

3.4.1.4 Heavy scale

Thick or adherent scale shall be removed by wet or dry blasting depending on dimensional tolerances before polishing per Specification MS 150.1.

3.4.2 Cleaning

3.4.2.1 Scale removal

When heat scale exceeds that encountered during spot welding it shall be removed by wet or dry abrasive means per Specification MS 150.1. The grain size of the grit employed must be consistent with the dimensional tolerances of the part and its finish specification.

3.4.2.2 Degrease

Parts shall be vapor or liquid degreased per Specification MS 150.0.

3.4.2.3 Alkaline clean

Parts cleaned per Specification MS 150.0 shall be further cleaned by an immersion in an aqueous solution containing 4 to 10 ounces per gallon of Wyandotte B. N. or equal and at a temperature of 190° to 210° F. Parts shall be immersed for 2 to 20 minutes depending upon the degree of soil.

3.4.2.4 Electroclean

Parts shall further be cleaned by electrolyzing an aqueous solution of Wyandotte B. N. or equal at a temperature of 140° to 160° F and at a

concentration of 4 to 6 ounces per gallon. The work shall be cathodic to avoid passivation, and a current density of 20 to 50 amperes per square foot shall be used. The time of immersion shall be from 5 to 7 minutes.

3.4.2.5 Hot rinse

Parts shall be rinsed in water at 150°F ($\pm 10^\circ$) and spray rinsed upon removal from the tank.

3.4.2.6 Cold rinse

Parts shall be rinsed in water at 150° F ($\pm 10^\circ$) and spray rinsed upon from the tank.

3.4.2.7 Electropolish

A. Racking - Parts shall be racked to provide effective, ample, electrical contact and to permit free evolution of gas without the danger of gas pockets.

B. Immersion - While power is disconnected, parts shall be immersed approximately 3/4 inch below the surface of the bath. The bath shall be as required in paragraph 3.2.5, and it shall be operated at 170° to 190° F for Type 300 series stainless steels and at 190° to 215° F for other steels. A current density of 0.5 to 1.25 amperes per square inch is required with this bath. Parts shall be immersed from 5 to 15 minutes depending upon the degree of polishing desired and upon whether the part is to be plated (5 before plating).

C. Agitation - To prevent gas streaks and pitting caused by fixation of bubbles, the bath should be agitated by air or by mechanical means.

D. Foam control - To avoid the entrapment of hydrogen released at the cathode, defoaming agents such as Colgate-Palmolive-Peet Company's "Vel" or equal may be added as required.

E. Anode to cathode spacing - The distance may vary from 2 to 8 inches depending upon the desired uniformity of etching. The cathode to anode area should be approximately two to one with the greatest area of the part parallel to the cathode.

F. Etch rate - Under normal operating conditions the rate of metal removal for the solution in paragraph 3.2.5 will be 0.0001 inch per minute. This rate will be increased with higher current densities.

3.4.2.8 Hot rinse No. 1

After power is disconnected, remove parts from electropolishing bath, allow to drain well, and rinse by immersion in water at 150° F ($\pm 10^\circ$) for 5 minutes. Spray rinse the part when removing it from the rinse. This tank should preferably possess a dam to allow constant overflowing and be emptied daily.

3.4.2.9 Hot rinse No. 2

Using water at 150° F ($\pm 10^\circ$) either immerse the part or spray rinse it thoroughly to remove the last traces of electropolishing acids.

NOTE: This is especially necessary where parts are to be plated. Chromic acid present in one part per million causes plating to only partially adhere. Chromic acid is a common ingredient of many electropolishing baths.

3.4.2.10 Dry

Parts shall be air dried or dried by blasting with pressurized air to facilitate drying.

3.4.2.11 Activating bath

When parts are to be plated following electropolishing, they shall be processed quickly through the two rinses and transferred, wet to the activating bath to avoid self-passivation.

A. Activation bath conditions - The bath shall be composed of an aqueous solution of 32 ounces per gallon of nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$), commercial grade, and 16 ounces per gallon of concentrated hydrochloric acid. It shall be operated at room temperature and the time of immersion shall be 30 seconds. A current density of 150 amperes per square foot shall be used with the work as the cathode and bagged, depolarized nickel as the anode.

3.4.2.12 Rinse

Parts shall be rinsed in cold, flowing water following activation and transferred wet to the plating bath. The pH of this rinse water shall be 2.5 to 3.5 and it shall be maintained by additions of hydrochloric acid.

NOTE: If 4 hours have elapsed between activation and plating, the parts shall be reactivated and rerinsed.

3.4.2.13 Plating

Electrodeposited coatings shall be in accordance with appropriate specifications, directives, or contractual agreements.

4. MANUFACTURING CONTROL PROVISIONS

4.1 General

Electropolishing shall be controlled by visual inspection. The finish shall be devoid of pitting, matting with blue tinge, brown opalescence, yellow spots, white streaks, dark brown spots, silver-white spots, bluish-brown tinge after an alkali treatment following electropolishing or waviness near holes. When parts prove unacceptable, they shall be reprocessed, if dimensions permit.

4.2 Viscosity

Viscosity of the electropolishing bath is an important control factor. As the bath ages, viscosity rises with the increasing solution of salts formed in the process. It will drop if the sulphuric acid content is not replenished as needed. Viscosity control in conjunction with regular, weekly, chemical analyses is the most effective means to successful polishing.

4.3 Passivity

Since an amorphous chromium-iron-oxygen film is formed in electropolishing, samples of the material polished must withstand the salt spray test in Standard 151A for 48 hours.

4.4 Electropolishing Solution Control

4.4.1 Water content

While water is lost by evaporation, the addition of water should be followed by increasing the bath temperature to 190° F ($\pm 10^\circ$). Water content

must be limited as brightness will diminish and etching sets in. It increases solubility of the products of the anode and facilitates diffusion of acid to the film. This causes an increase in current density and resultant passivation.

4.4.2 Iron content

Since it is not economically practical to plate out the iron content of the bath, its concentration must be observed because at 6 to 7 percent ferric oxide the bath becomes ineffective. Also at 2.50 to 3.0 percent ferric oxide, the current density must be altered to obtain a satisfactory finish.

4.4.3 Chromic ion

In electropolishing solutions containing chromic acid, the hexavalent chromium ion, that is reduced to the trivalent ion by the hydrogen at the cathode increases the viscosity of the bath and decreases the conductivity of the solution.

4.4.3.1 Oxidation of trivalent chrome to hexavalent chrome

Electrolyzing the solution with lead anodes and cathodes will cause the anodic oxygen to oxidize the chromium ion to the hexavalent state. The lead chromate formed at the cathode can be forestalled by the use of perforated ceramic diaphragms. At the end of the chromium ion oxidation heat the solution to 200° F for 1 hour and allow it to stand at room temperature for 1 hour.

4.4.4 Acid content

Acids shall be added as chemical analyses dictate. Glycolic acid may be allowed to decrease to 50 percent, but the sulfuric acid should be held near 30 percent. The usual consumption ratio is about three parts sulfuric acid to one part of glycolic acid (pure acid base). Acid content is depleted by combination with ferric and chromic ions, by drag-out, by mist formation, and by oxidation of glycolic acid to formaldehyde.

4.4.5 Sludging

Sludge formed in the bath or upon cathodes should be removed periodically to prevent increasing polarization voltage and inefficient bath conditions.

4.4.6 Life of bath

Since dissolved metal content varies with the sludging or nonsludging characteristics of the bath, some baths never exceed 2 percent, while others will contain up to 6 percent dissolved metal. Thus each bath has a final working life.

4.5 Control of Other Solutions

Solutions used in cleaning, rinsing, activating, and cleanliness preservation shall be analyzed as required to effectuate successful electropolishing.

5. PREPARATION FOR DELIVERY

5.1 General

The protection given parts during handling and shipment must be ample and adequate to ensure acceptable parts. When parts for LOX service are involved, they shall be treated as required by purchasing agreements, drawings, or related specifications.

6. NOTES

6.1 Intended Use

This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use when electropolishing corrosion-resistant steel for use in space launch vehicle components.

6.2 Caution

Surface ventilation at the electropolishing tank is necessary to remove the spray that forms, the hydrogen that is evolved, and the formaldehyde fumes that occur when overheating at an electrode exists. Defoaming agents are recommended to avoid hydrogen entrapment and the potential explosion hazard, if arcing should occur.

NOTICE: When government drawings, specification, or other data are used for any purpose other than in connection with a definitely related government procurement operation, the United States Government incurs no responsibility nor any obligation; and the fact that the Government may have formulated,

furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

CLEANING MISCELLANEOUS MATERIAL/COMPONENTS
MANUFACTURING SPECIFICATION FOR
CLEANING OF CONTROL ASSEMBLIES (VALVES)

1. SCOPE

1.1 Scope

This specification covers the approved PE Laboratory manufacturing requirements for the cleaning of control assembly (valve) components for use in space launch vehicles.

1.2 Applicability

The cleaning requirements set forth in this manufacturing specification are applicable to control assembly components fabricated from the following materials.

- Stainless steels, Types 302, 304, 304L, 316, 316L, 321, and 347
- Iron, Armco (Copper-Nickel-Chrome Plated)
- Aluminum alloys, 2024, 6061, 1100, 5052
- Inconel
- Glass Melamine
- Nylon (SP-126-2) or FM 3001 Natural
- Rubber, Buna N, or Silicone
- Teflon
- Allpax 500
- Kel-F

2. APPLICABLE DOCUMENTS

2.1 Governmental

The following documents, of the issue in effect on the date of use of this specification, form a part of this specification.

SPECIFICATIONS

Federal

O-P-94A	Paper, Test; pH Indicating
O-T-634a	Trichloroethylene, Technical
BB-N-411A	Nitrogen
QQ-A-267(2)	Aluminum Alloy 24S, Bars, Rods, and Shapes, Extruded
QQ-A-268(1)	Aluminum Alloy 24S Bars, Rods, and Wire (Rolled or Drawn)
QQ-A-270A	Aluminum Alloy Bars, Rods, and Shapes, Extruded, 6061 and 6062
QQ-A-315A(1)	Aluminum Alloy 52S Bars, Rods, and Wire (Rolled or Drawn)
QQ-A-318C	Aluminum Alloy Plate and Sheet 5052
QQ-A-325A(2)	Aluminum Alloy 61S Bars, Rods, Shapes, and Wire (Rolled or Drawn)
QQ-A-561C	Aluminum Alloy 1100 (2S) Plate and Sheet
QQ-C-320(1)	Chromium Plating (Electro deposited)
QQ-S-763B(1)	Steel Bars, Shapes, and Forgings, Corrosion Resisting
UU-T-81F(1)	Tags, Shipping, and Stock
ZZ-G-421a(5)	Gloves, Rubber, Surgeons

PPP-T-60(3) Tape, Pressure Sensitive Adhesive,
Waterproof, for Packaging and Sealing

Military

MIL-A-00148B Aluminum Foil

MIL-P-3086 Plastic Material, Thermoplastic,
Nonrigid, Polyamide Resin (Nylon)

MIL-R-5847C Rubber, Silicone, High and Low
Temperature Resistant

MIL-P-15037C Plastic Material, Laminated,
Thermosetting, Sheets, Glasscloth,
Melamine Resin

MIL-D-16791 C Detergent, Nonionic

MIL-R-25988(USAF) (1) Rubber, Silicone, Oil and Fuel
Resistant

George C. Marshall Space Flight Center

MSFC-SPEC-164 Cleanliness of Components for Use in
Oxygen, Fuel, and Pneumatic Systems,
Specification for

MS 101.0 Manufacturing Specification for The
Drying of Components in a Vacuum

2.2 Other Publications

The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

Society of Automotive Engineers, Inc.

AMS 3649A Polytrifluorochloroethylene - Film
Unplasticized (Kel-F)

AMS 3650A	Polytrifluorochlorethylene - Unplasticized (Kel-F)
AMS 3651B	Polytetrafluoroethylene (Teflon)
AMS 3652	Polytetrafluoroethylene Film (Teflon)
AMS 5699A	Wire, Round, Nickel Base - 15.5 Cr-7 Fe-2.3 Ti-1 (Cb + Ta) - 0.7Al, Spring Temper (Inconel X)
AMS 7272	Rings Sealing, Synthetic Rubber, Synthetic Lubricant Resistant, Buna-N Type
AMS 7706	Iron, Commercially Pure - Hot Rolled, Annealed - Plate, Sheet, Strip, and Bar
AMS 7707	Iron, Commercially Pure - Hot Rolled, Unannealed - Plate, Sheet, Strip, and Bar

(Copies of the publications listed above may be obtained from the American Society of Automotive Engineers, Inc., 485 Lexington Avenue, New York 17, New York.)

The Allpax Company, Inc.

Allpax 500

(Copies of the publication listed above may be obtained from The Allpax Company, Inc., 160 Jefferson Avenue, Manaroneck, New York.)

3. REQUIREMENTS

3.1 General

No deviation from the requirements of this manufacturing specification shall be permitted without prior written approval of the MSFC initiating activity.

3.2 Materials

3.2.1 Trichloroethylene for vapor degreasing

The trichloroethylene used for vapor degreasing, under the provisions of this specification, shall meet all of the requirements for Type II of Specification O-T-634a.

3.2.2 Trichloroethylene for cold degreasing

The trichloroethylene used for cold degreasing, under the provisions of this specification, shall meet all of the requirements for Type I of Specification O-T-634a, except that the nonvolatile residue content shall not be greater than 0.010 gram per 500 ml when tested as specified in paragraph 4.4.2.2 of Specification MSFC-SPEC-164. In addition, 500 ml samples shall not contain a particle larger than 175 microns in any dimension or more than 5 particles between 100 and 175 microns in any dimension.

3.2.3 Nonionic detergent

The nonionic detergent used for cleaning under the provisions of this specification shall meet all of the requirements for Type II of Specification MIL-D-16791C. (The only detergent now approved for use is Dowell F-33.)

3.2.4 Demineralized water

The minimum specific resistance of demineralized water shall be 50 000 ohms. It shall contain no particles larger than 175 microns in any dimension.

3.2.5 Drying or preservation gases

Air or nitrogen used in drying or preserving of control assembly components shall contain no particles larger than 100 microns (absolute), the oil content of the gases shall not be greater than 3 parts per million (by weight), and the moisture content of the gases shall not be greater than 24 parts per million (by volume).

3.2.5.1 Nitrogen

Nitrogen gas, to be used in drying or preservation processes, shall

conform to Type I, Class I, Grade B of Specification BB-N-411a, except that the particle size, oil content, and moisture content requirements of 3.2.5 shall be met.

3.2.5.2 Air

Compressed air used in drying or preservation processes shall meet the particle size, oil content, and moisture content requirements of paragraph 3.2.5 above.

3.2.6 Paper, pH indicating

The pH indicating paper used in determining the acidity or alkalinity of control assembly component surfaces shall be of a grade conforming to Specification O-P-94a.

3.2.7 Aluminum foil

Dry annealed aluminum foil used in the protection of cleaned control assembly components shall conform to Specification MIL-A-00148C.

3.2.8 Tape

The tape used to secure protective coverings on cleaned control assemblies or components shall conform to Specification PPP-T-60(3).

3.2.9 Tags

The tags used to mark and identify cleaned control assemblies or components shall conform to Specification UU-T-81F(1).

3.3 Level of Cleanliness and Dryness

Control assemblies or control assembly components cleaned by the procedures outlined in this manufacturing specification shall meet all of the particle size, nonvolatile residue, and dryness requirements of Specification MSFC-SPEC-164.

3.4 Cleaning of Metallic Control Assemblies and Their Components (Including Teflon, Kel-F or Combinations of Teflon or Kel-F with Metallic Parts)

3.4.1 Oxygen, fuel, and pneumatic system control assemblies (valves)

3.4.1.1 Completely disassemble the control assembly into all of its individual components.

3.4.1.2 Ultrasonically clean all the the control assembly components in an ultrasonic cleaning tank utilizing a cleaning solution of 5 parts nonionic detergent (by volume) to 1000 parts of demineralized water. Clean for 5 to 10 minutes at a solution temperature of 120° F ($\pm 10^\circ$).

3.4.1.3 Using clean rubber surgical gloves, transfer the ultrasonically clean components from the detergent cleaning bath to a demineralized water rinsing bath. All handling of cleaned components from this point until control assemblies are reassembled and packaged shall be with clean rubber surgical gloves.

3.4.1.4 Thoroughly rinse the cleaned control assembly parts by flushing with or immersing in demineralized water at ambient temperature for 3 to 5 minutes. After rinsing, the used rinse water or effluent shall register a pH within the range of 6 to 8 when contacted by pH indicating paper.

3.4.1.5 After thorough rinsing, a 500 ml sample of demineralized water shall be flushed over or through the surfaces which will be in contact with oxygen, fuel, or pneumatic environments. This sample shall be checked for particle size in the manner specified in paragraph 4.4.2.1 of Specification MSFC-SPEC-164. Particles of contamination per square foot of control assembly component surface area tested shall not exceed the following limits.

- No particle larger than 2500 microns in any dimension.
- One particle between 700 and 2500 microns in its largest dimension.
- Five particles between 175 and 700 microns in their largest dimension.

3.4.1.6 Thoroughly dry the rinsed control assembly parts by blowing with compressed air or nitrogen, or by heating in a vacuum oven as specified in Specification MS 101.0. This drying step completes the cleaning procedure for fuel and pneumatic system control assemblies and components.

3.4.1.7 For oxygen valve components, additional cleaning as specified in 3.4.2 below is required.

3.4.1.8 At this point all other components are clean and ready for reassembly and pressure test.

3.4.1.9 Protect control assembly with a minimum of two layers of aluminum foil secured with tape. The assemblies or components shall then be placed in clean polyethylene bags, the bags purged with nitrogen or air, the bag heat sealed, and packed in a manner that will prevent damage during storage or handling.

3.4.2 Oxygen system control assemblies (Cleaning procedure continued from 3.4.1.6 above)

3.4.2.1 Ultrasonically degrease all of the control assembly components in an ultrasonic cleaning tank for 5 to 10 minutes utilizing Type I trichloroethylene at ambient temperature as the cleaning medium. Use gloves and respirators, unless a fume exhaust hood or room exhaust system is available for this operation.

3.4.2.2 Thoroughly rinse the degreased components with new Type I trichloroethylene at ambient temperature for 3 to 5 minutes. Use gloves and respirator, unless fume exhaust hood or room exhaust system is available for this operation.

A sample of the rinse trichloroethylene shall be checked for non-volatile residue pickup in the manner specified in 4.4.2.2 of Specification MSFC-SPEC-164. The nonvolatile residue contamination on the surfaces to be contacted by liquid or gaseous oxygen shall not be greater than 0.001 gram per square foot.

3.4.2.3 Thoroughly dry the degreased control assembly components by blowing with compressed air or nitrogen, or by heating in a vacuum oven as specified in the Specification MS 101.0.

3.4.2.4 At this point components are clean and ready for reassembly and pressure test.

3.4.2.5 Protect control assemblies from contamination by covering all openings into the control assembly with a minimum of two layers of aluminum foil secured with tape. The control assemblies or components

shall then be placed in clean polyethylene bags, the bags purged with nitrogen or air, bag heat sealed and packed in a manner that will prevent damage during storage or handling.

3.5 Cleaning of Nonmetallic Components (Including Nonmetallics, Except Teflon or Kel-F, Permanently Attached to Metal)

3.5.1 Cleaning

Immerse each component in a solution of 5 parts nonionic detergent (by volume) in 1000 parts demineralized water. Clean for 10 to 15 minutes by gently scrubbing the part with a nylon bristle brush at a solution temperature of 110° F ($\pm 10^\circ$).

3.5.2 Handling

Using clean rubber gloves, transfer the cleaned components from the detergent cleaning bath to a demineralized water rinsing bath. During the remainder of the cleaning procedure, all handling of cleaned components shall be with clean rubber gloves.

3.5.3 Rinsing

Thoroughly rinse the cleaned components by immersion in or flushing with demineralized water at ambient temperature for 3 to 5 minutes.

3.5.4 Drying

Thoroughly dry the rinsed components by blowing with compressed air or by heating in an oven at 110° F ($\pm 10^\circ$) for a minimum of 30 minutes.

3.5.5 Protecting

Protect the dried components from recontamination before valve reassembly by wrapping them in a minimum of two thicknesses of aluminum foil secured with tape.

3.6 Recleaning Procedures

Any control assembly, rejected and requiring recleaning, will be disassembled and recleaned in accordance with paragraphs 3.4 and 3.5. Any control assembly components (metallic and nonmetallic), after original cleaning cycle for each component as follows.

- Most control assemblies, once removed for inspection, require new gaskets, and/or "O" ring or washers and therefore require a full cleaning.

- The operations outlined in their original cleaning specification are not lengthy and none are damaging to components, even if repeated numerous times.

- A lowering of cleaning requirements always results in a corresponding drop in the level of quality.

4. QUALITY ASSURANCE PROVISIONS

4.1 Acceptance Inspections

4.1.1 Visual examination

All cleaned control assembly components shall be visually inspected for the presence of moisture and foreign materials such as corrosion, scale, dirt, oil, grease, and similar foreign materials. The presence of visible contamination shall be a cause for rejection and will necessitate recleaning of the component. Scale-free discoloration due to welding is permitted.

4.1.2 Surface acidity or alkalinity

Surfaces of cleaned and rinsed control assembly components shall be tested with pH indicating paper while the components are still wet from the last rinse. Surfaces shall register a pH between 6 and 8.

4.1.3 Dryness

Surface dryness of cleaned, rinsed, and dried control assembly components shall be determined by passing dry gas through or over the surfaces of the component.

4.2 Reliability Tests

4.2.1 Particle size

Reliability test samples for particle size shall be prepared as specified

in 4.4.1 of Specification MSFC-SPEC-164 and tested as specified in 4.4.2.1 of Specification MSFC-SPEC-164.

4.2.2 Nonvolatile residue

Reliability test samples for nonvolatile residue determination shall be prepared as specified in 4.4.1 of Specification MSFC-SPEC-164 and tested as specified in 4.4.2.2 of Specification MSFC-SPEC-164.

5. PREPARATION FOR DELIVERY

5.1 Preservation and Packaging

Control valve components for use in space launch vehicle shall be packaged as specified immediately upon completion of the assembly operation in order to prevent contamination. The control assemblies shall be packed in containers capable of providing protection against damage during storage, handling, or transportation before installation.

5.2 Marking

Cleaned and packaged control assemblies shall be marked with tags, conforming to Type C of Specification UU-T-81F(1), which shall contain the following information.

- Part or identification number
- Method of cleaning and micron level
- Date of cleaning
- Title, date, and number of this manufacturing specification
- Contractor identification
- Manufacturer's serial number
- Service medium or intended use of control assembly

6. NOTES

6.1 Intended Use

This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use in the cleaning and preservation of control assembly (valve) components for use in oxygen, fuel, or pneumatic systems.

NOTICE: When government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related government procurement operation, the United States Government incurs no responsibility nor any obligation; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING PROCESS FOR CLEANING
AND TESTING OF GAS BEARING GAS SUPPLY
SYSTEM COMPONENTS AND HELIUM SLOSH MEASURING
SYSTEM COMPONENTS FOR SPACE VEHICLE USE

1. SCOPE

1.1 Scope - This manufacturing process covers the approved Process Engineering Laboratory requirements for the cleaning, processing, and testing of gas bearing gas supply system components and helium slosh measuring system components for space vehicle use.

1.2 Applicability - The requirements set forth in this process are applicable to:

1.2.1 Nonmetallic material such as synthetic rubber, phenolic, acrylic, nylon, mylar, Viton A "O" rings, glass melamine, synthetic resin, teflon, poroloy, and cerameterm or nonmetallic combined with metallic materials.

1.2.2 Metallic material such as aluminum 380, 2SH19, 356T-6, 3003, 2017T4, 2024T4, 5052, 6061T6, 7075T6; stainless steel 18-8 cres, 17-4 pH, 17-7 pH cres, 302 cres, 3035 C cond. A, 301, 303 cres, 302 cond. B, 303 cond. A, 304, 316 cres, 347, 440 cres; Chromel A (nickel-chromium) copper, tin, brass, silver, titanium, bronze, nickel, and cadmium.

1.2.3 Coated metallic material - anodized and electroplated metallic material.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this process, form a part of this process.

SPECIFICATIONS

Federal

O-E-760

Ethyl Alcohol (Ethanol); Denatured Alcohol; and Proprietary Solvent.

O-T-634A	Trichloroethylene, Technical.
BB-N-411	Nitrogen.
O-A-51C	Acetone, Technical.

Military

MIL-D16791	Detergent, Nonionic.
O-P-94	Paper, Test, pH Indicating.

George C. Marshall Space Flight Center

MS 100.4	Manufacturing Specification for the Cleaning and Passivation of Austenitic (300 Series) Stainless Steel Suction Lines.
MS 100.7	Manufacturing Specification for the Cleaning of Control Assemblies (Valves).
MS 100.8	Cleaning of Flared Tubing.
MS 100.9	Cleaning of Miscellaneous Components.
MS 101.0	Drying Components in a Vacuum
MS 150.0	Vapor degreasing of Metallic Surfaces.
MSFC 10419906	Specification for Cleanliness Levels, Cleaning and Inspection Procedures for Component Parts of Gas Bearing and Sloss Measuring Systems.
M-ME-MPROC 150.8	Manufacturing Specification for the Cleaning of Spheres for the C-1 Space Vehicle.
MSFC-SPEC-237	Solvent "Freon" Precision Cleaning Agent.

2.2 Other Publications

Air Force TO 00-25-203 Standard Functional Criteria for
Design and Operation of Clean Rooms.

(Copies of above publication may be obtained from USAF activities, in accordance with T. O. 00-5-2 U. S. Air Force)

Technical Bulletin
S-FST-2

Freon Precision Cleaning Agent

(Copies of above publications may be obtained from E. I. Dupont De Nemours & Company, Inc., Wilmington 98, Delaware)

No Publications
Available

Cambridge Filter Corp. Catalog

(This catalog may be obtained from the Cambridge Filter Corporation, 738 E. Erie Blvd., Syracuse, N. Y.)

3. REQUIREMENTS

3.1 General - No deviation from the requirements of this manufacturing process shall be permitted without prior written approval of the Process Engineering Laboratory.

3.2 Materials

3.2.1 Testing Solvents

3.2.1.1 Ethyl Alcohol - Ethyl alcohol conforming to Grade I, Class B; Grade II, Class B; or Grade III of Specification O-E-760 shall be used for testing the surfaces of nonmetallic (except teflon) components for cleanliness. Before use the alcohol must be filtered through 42 thicknesses of Whatman, Number 42 filter paper, or an equivalent, to remove particles greater than 20 micron in size in any dimension.

3.2.1.2 Trichloroethylene - Trichloroethylene, Type I, conforming to Specification O-T-634, shall be used for testing the surfaces of metallic and teflon components for nonvolatile residue cleanliness, except that the non-volatile residue of the solvent shall not be greater than 0.002 g per 100 ml.

Before use, the trichloroethylene must be filtered through Whatman No. 42 filter paper, or an equivalent, to remove particles greater than 20 microns in size in any dimension.

3.2.1.3 "Freon," precision cleaning agent - The "Freon," precision cleaning agent, conforming to MSFC-SPEC-237, shall be used for testing the surface of metallic and teflon components for nonvolatile residue cleanliness, except that the nonvolatile residue of the solvent shall be not greater than 0.002 g per 100 ml. Before use, the "Freon," precision cleaning agent must be filtered through Whatman No. 42 filter paper, or an equivalent, to remove particles greater than 20 microns in size in any dimension.

3.2.2 : Other Materials

3.2.2.1 Demineralized Water - Demineralized water, if required for rinsing operations, shall be prefiltered to remove all particles greater than 100 microns in size in any dimension. The minimum specific resistance of the demineralized water shall be 50 000 ohm.

3.2.2.2 Detergents - Detergent cleaning agents for use in cleaning processes shall conform to Specification MIL-D-16791 (Type I, water soluble).

3.2.2.3 Packaging materials - Materials used to package gas bearing components or assemblies shall be precleaned to meet the particle requirements of Drawing No. 10419906. (Refer to Saturn Packaging Manual.)

3.2.2.4 Filter paper - Filter paper shall be Whatman No. 42, or equivalent.

3.2.2.5 Drying and preservation gases - Gases used in drying and preservation processes shall be checked for hydrocarbons, moisture content, and particulate contamination. Hydrocarbon content shall not exceed 0.3 part per million (by weight). The dewpoint shall be -65°F maximum. Particle size shall not exceed 20 microns in any dimension.

3.2.2.6 Nitrogen - Nitrogen gas used for drying or preservation processes shall conform to Type I, Class 1, Grade A of Specification BB-N-411, except that requirements of paragraph 3.2.2.5 shall be met.

3.2.3 Levels of Cleanliness

3.2.3.1 Nonmetallic materials (except teflon) - Nonmetallics shall be free of particles greater than 20 microns in size in any dimension and shall be visibly free of contamination, such as oil or greases.

3.2.3.2 Metallic materials and teflon

A. Metallic and teflon material shall be free of particles greater than 20 microns in size in any dimension.

B. When testing surface areas of 1 square foot or less, as specified in paragraph 4.1.3.1.2 of Drawing No. 10419906, the difference between the used and unused testing solvent shall indicate an increase of not more than 0.0005 gm per 100 ml in the nonvolatile residue content of the used solvent.

C. When testing surface areas greater than 1 square foot, the increase shall not be more than 0.001 gram per square foot.

3.2.3.3 Coated metallic materials - Same as paragraph 3.2.3.2.

3.3 Work Area

3.3.1 Facilities

3.3.1.1 Precleaning facilities

A. Vapor degreaser tank

- The temperature of this tank will be 188°F ($\pm 3^\circ$).
- A spray lance will be readily accessible for use in spray degreasing.

B. Ultrasonic detergent tank

- This tank will be made of stainless steel and will be 4 ft long, 3 ft wide, and 3 ft deep.
- A steam heat exchanger will be located within the tank, and a temperature regulator will be capable of maintaining temperatures from ambient to 180°F ($\pm 10^\circ$).
- The ultrasonic circulation system will be capable of supplying filtered deionized water to 100 microns (absolute).

C. Trichloroethylene rinse tank

- This tank will be made from stainless steel and will be 4 ft. long, 3 ft wide, and 3 ft deep.

- A 75 micron filter will be between storage tank and rinse tank.
- Provisions will be made for a recirculation rinse through the tank or through a spray nozzle attached to the tank.

D. Rinse tank

- This tank will be made from stainless steel and will be 4 ft long, 3 ft wide, and 3 ft deep.
- Provisions will be made for a spray or submerged rinse with agitation.
- The water will be agitated with filtered missile grade air at the bottom of the tank.
- The water in the tank will be deionized and filtered to 100 microns (absolute).

3.3.1.2 White Bench System (Matthews Research, Inc. Bench) - A facility which incorporates high standards of environmental control and cleanliness necessary to meet particular requirements of a Class IV clean room (AF-TO-00-25-203) will be used for cleaning, checking, assembling, functional testing, and packaging.

A. Auxiliary facilities

- Air-conditioning will be provided to control the temperature at 72°F ($\pm 1^\circ$) and the relative humidity at 45 percent maximum. The bench is located in a room at 72°F ($\pm 5^\circ$).
- The solvent circulation system shall be capable of supplying filtered solvent to 10 microns absolute.
- Two vacuum pumps, one for each oven, will be supplied. These pumps will be capable of producing a vacuum of 25 inches Hg.
- The electrical system will operate from 110/220 volts.
- A function test panel will operate a source of pneumatic pressures from 0 to 3000 psig directly to the work benches.

- Ultrasonic generators will produce the power for ultrasonic cleaning.

B. Cleaning bench

- A transparent enclosure presenting a positive barrier to airborne contamination shall cover the bench.
- An air lock will be located at the entrance of this bench.
- A vacuum oven will be at the exit to the bench. This oven will be capable of 25 inches Hg vacuum with temperature control of ambient to 250°F ($\pm 5^\circ$).
- The surface will be lighted evenly to a minimum level of 175 ft candles.
- This bench will contain 120 volt outlets.
- Two 20 gallon ultrasonic tanks will be built into the bench. These tanks will be used separately.
- Two 15 gallon rinse tanks will be located within this bench.
- A vent will be located at the top and bottom of the enclosed bench so fumes may be exhausted.
- The capability of working from both sides of this bench will exist.

C. Checking bench

- For similar details of this bench, see paragraph 3.3.1.2.B.
- The bench will have a smooth glare free surface 12 feet long and 34 inches wide.
- The vacuum oven at the exit of the cleaning bench will open into the checking bench. Another vacuum oven will be at the exit of this bench.

D. Assembly bench

- For similar details of this bench see paragraphs 3.3.1.2.B and 3.3.1.2.C.
- The vacuum oven at the exit of the checking bench will open into the assembly bench. An air lock will be at the exit of this bench.

E. Functional testing and packaging bench

- For similar details of this bench, see paragraphs 3.3.1.2.B and 3.3.1.2.C.
- Within the air lock at the entrance of this bench, the capability of ultrasonically cleaning tools as they are introduced into the bench will exist. Refer to 3.4.7.1.B for precleaning of the tools before entering air lock.
- An air lock will be at the exit of this bench.

3.3.1.3 Whitfield Cleaning Bench System

A. Whitfield bench

- This bench will be approximately 4 feet long, 4 feet wide, and 3 feet high.
- Spheres and tubing will be cleaned in this bench.
- A double sink, 18 x 18 x 9 inches, will be used to water rinse parts.
- The air filtering system will consist of two rough filters and a cambridge filter.
- This bench will have a self-enclosed blower unit.

3.3.1.4 Liquid Cleaning Console - The liquid cleaning console will serve to supply ultra clean (10 micron absolute) fluids to both the Matthews and Whitfield bench systems.

A. The cleaning agent will be either alcohol, "Freon" Precision Cleaning Agent, or trichloroethylene.

B. Pressurized filtration of the liquid through a series of filters will take place in welded stainless steel pressure vessels.

C. A hand operated valve is installed to facilitate the taking of samples.

D. Air or nitrogen, used for purging, is filtered through three different filters after entering the console to reduce particle size to 2 microns maximum.

3.3.2 Equipment

3.3.2.1 Bench systems

A. Particle size test equipment

- One-liter vacuum flash.
- Forceps with unserrated tips.
- Plastic disposable Petri dishes.
- Aspirator or other suitable source of vacuum.
- Microscope with mechanical stage, measuring eyepiece with ocular micrometer, and capability of magnification of 45X and 90X minimum.
- Millipore filter holder assembly - Cat. No. XX 1004700 or equivalent.
- Plastic filter paper holder.
- Microscope lamp 5000 candle power minimum.
- Whatman 42 filter paper or approved equivalent.
- Wash bottle.

3.3.3 Accessory Equipment

3.3.3.1 Dust preventive clothing (smocks, coveralls, caps, gloves, and boots) shall be worn by personnel while working in cleanroom or clean bench area.

3.3.3.2 Portable test equipment, jigs, fixtures, parts, and subassemblies which, due to size, material composition, or critical finishes, which cannot be precleaned per paragraph 3.4.7.1.B will be thoroughly vacuumed and covered with lint-free non-shedding plastic covers prior to entering clean bench.

3.3.3.3 All materials shall be taken into the clean bench through an air lock.

3.3.3.4 Movement of calibration and support equipment, and similar equipment, in and out of the clean bench shall be reduced to a minimum. Care shall be taken to insure that these items are thoroughly vacuumed before entering clean bench.

3.3.3.5 Partly completed work shall be protected by covering with suitable covers of metal or plastic. Plastic bags or molded, chip proof plastic trays, and covers are particularly well suited for protection purposes. This equipment will prevent contamination with unfiltered particles which may be settling through the air in the clean bench.

3.3.3.6 Millipore filter - The millipore filter paper, 47 mm diameter .045-micron membrane, is type HA black imprinted grid on 3.08 mm centers with each square equal to 1/100th of the total effective filtering area of the disc.

3.3.3.7 Stainless steel mesh 2-micron filter - This filter is a line type which can be serviced without disturbing system connections. It is a porous metal filter media type used in series with the 5 micron fibrous filter type. (Purolator Products Inc., Rahway, N. J.)

3.3.3.8 Paper 5-micron filter - This filter is a fibrous type filter media used in series with the 2 micron porous metal filter. (Purolator Products, Inc., Rahway, N. J.).

3.3.3.9 List of nonvolatile residue equipment

- 800 ml beaker
- steam bath
- weighing bottle
- analytical balance
- constant temperature oven
- desiccator

3.4 Procedure No. 1

3.4.1 Metal, Teflon, and Plated Components (Valves, Thermistor, etc.)

3.4.1.1 Disassembly - Assemblies shall be disassembled into components where applicable.

3.4.1.2 Precleaning - Preclean uncoated, metallic and teflon components as shown below. No testing is required in all cases; however, drying of components is required. No packaging is required if a delay of no more than 1 or 2 hours is incurred between precleaning and final cleaning operations. If more than 2 hours delay is incurred, cover the components in covered plastic containers or polyethylene bags.

A. Vapor degrease - Vapor degrease the parts by lowering into the trichloroethylene vapors. Leave the parts in the vapors until condensation on the surface ceases.

B. Liquid degrease - Flush or circulate trichloroethylene, at ambient temperature, over or through the surfaces of the components for 10 to 30 minutes. For items requiring removal of varnish type, organic material from the surface immerse the part in acetone for 10 to 30 minutes.

NOTE: This operation shall be performed in a well ventilated area which shall be free of any possible source of ignition such as static electricity, electric motors, switches, smoking, and open flame. An exhaust hood should be used when available.

C. Dry - Dry the parts with drying gas (para. 3.2.2.5) at 180°F (±20°) or dry in a vacuum oven or a drying oven.

D. Ultrasonic clean - Place the part in an ultrasonic bath consisting of 5 parts detergent to 1000 parts of water.

E. Spray rinse - Spray rinse, flush, or immerse with distilled or demineralized water for 10 to 15 minutes of 90°F ($\pm 20^\circ$) or until all sudsing has ceased.

F. Dry - Components shall be dried using a gas meeting the requirements of 3.2.2.5, or in a vacuum oven at 180°F ($\pm 20^\circ$) at 5 to 50 mm of Mercury for 1 hour. Items of Grade 300 series stainless steel which have light corrosion at this point shall be processed as specified in paragraph 3.4.1.2.7. Heavily corroded areas shall be cause for rejection of the part.

G. Phosphoric acid - Parts made of Grade 300 series stainless steel which demonstrate corrosion after processing through paragraph 3.4.1.2.F shall be immersed in a dilute 10 to 20 percent solution of inhibited phosphoric acid at ambient temperature in an ultrasonic bath for 5 minutes. Rinse and dry the part thoroughly as specified in paragraphs 3.4.1.2.E and 3.4.1.2.F.

3.4.1.3 Air lock - All components shall be taken into the clean bench through an air lock for the final cleaning operation.

3.4.1.4 Final cleaning - A Matthews Research bench employing cambridge absolute filter to filter the incoming air into the work space of the Matthews Research Inc. type work bench, or equivalent, shall be used in the final cleaning operation. The work bench temperature shall be 72°F ($\pm 5^\circ$) with a maximum relative humidity of 45 percent.

A. Metal (uncoated) - Metal components (uncoated) shall be ultrasonically cleaned utilizing a solution of 5 parts of detergent to 1000 parts of water conforming to Specification MIL-D-16791 (Type I-water soluble) for a period of 10 to 15 minutes maintained at a temperature of 130°F ($\pm 10^\circ$).

B. Rinse - Rinse component surfaces thoroughly with prefiltered demineralized water until suds free and then continue for a period of 5 to 10 minutes.

C. Dry - Dry component in a vacuum oven at 3 to 6 inches of mercury and 180°F ($\pm 20^\circ$).

D. Testing - A Matthews Research bench, or equivalent, with millipore filter equipment, or equivalent, for particle counts and nonvolatile residue sampling equipment shall be used in the testing operation.

- Metallic and teflon materials shall be free of particles greater than 20 microns in size in any dimension.
- When testing surface areas of 1 square foot or less, as specified in 3.5.1.C, the difference between the used and unused testing solvent shall indicate an increase of not more than 0.0005 g per 100 ml within 24 hours in the nonvolatile residue content of the used solvent over that of the unused solvent.
- When testing surface areas greater than 1 square foot the increase shall be not more than 0.001 g per square foot.

E. Drying - Immediately following cleaning or cleanliness tests, as applicable, the component shall be dried as specified in 3.4.1.D.

F. Assembly and packaging - A Matthews Research, Inc. type bench, or equivalent, shall be used for assembly of components.

- Materials and equipment used shall be as specified in 3.3.3 for assembly of components.
- Packaging of assemblies shall be accomplished in this operation in accordance with the instructions in the Saturn Packaging Manual.
- A Whitfield work bench, or equivalent, shall be employed for cleaning of packages. The packages shall be inverted over a spray nozzle or wand and flushed with ethanol or "Freon," which has been prefiltered to remove all particles greater than 20 microns in size in any dimension. The packages shall be dried as necessary. (Refer to the Saturn Packaging Manual.)

G. Air lock - Assemblies after packaging shall leave the assembly and packaging cabinet through an air lock.

3.5 Procedure No. 2

3.5.1 Coated Metal Components

3.5.1.1 Disassembly - Assemblies shall be disassembled into components.

3.5.1.2 Precleaning - Preclean coated metal components as outlined below (nonultrasonic).

A. Vapor degrease - Vapor degrease the parts by lowering into the trichloroethylene vapors. Leave the parts in the vapors until condensation on the surface ceases.

B. Dry - Dry the part in the manner specified in paragraph 3.4.1.2.F of this specification.

C. Wash - Scrub the surfaces of the component with a nylon brush saturated with 5 parts of Dowell F-33 detergent, or equivalent, in 1000 parts demineralized water.

D. Rinse - Rinse the part in demineralized water at 180°F ($\pm 20^\circ$) until suds-free and then continue to rinse for a period of 5 to 10 minutes.

E. Dry - Dry the part in the manner specified in paragraph 3.4.1.2.F of this specification.

3.5.1.3 Air lock - All components shall be taken into the clean bench through an air lock for the final cleaning operation.

3.5.1.4 Final cleaning - A Matthews Research bench employing Cambridge absolute filter to filter the incoming air into the work space of the Matthews Research Inc. type work bench, or equivalent, shall be used in the final cleaning operation. The work bench temperature shall be 72°F ($\pm 5^\circ$) with a maximum relative humidity of 45 percent.

A. Coated metal components - Coated metal components shall not be ultrasonically cleaned.

- Clean coated metal components by using a nylon bristle brush with detergent or with "Freon" as applicable.
- Rinse - Immersion rinse component surfaces thoroughly with prefiltered demineralized water until all sudsing ceases and for a period of 5 to 10 minutes if brushed with detergent only. If parts have been brushed with "Freon," they should be rinsed by immersion in "Freon."

- Dry - Dry component in a vacuum oven at 3 to 6 inches of mercury and 180°F ($\pm 20^\circ$).
- Testing - Testing is the same as specified in paragraph 3.4.1.D.
- Assembly and packaging - The assembly and packaging is the same as specified in Process No. 1. (Refer to paragraph 3.4.1.4.F.)

3.6 Procedure No. 3

3.6.1 Nonmetallic components

3.6.1.1 Disassembly - Assemblies shall be disassembled into components where applicable.

3.6.1.2 Precleaning - Preclean nonmetallic components, including metallic components combined with nonmetallic materials other than Teflon or Kel-F, by the following process.

A. Wash - Immerse spray (1 inch nozzle) or flush each part in a solution of 5 parts (by volume) detergent to 1000 parts of demineralized water for 10 to 15 minutes at a temperature of 110°F ($\pm 10^\circ$). When it is feasible, the careful use of a nylon bristle brush may assist in removing contaminants.

B. Rinse - Rinse by immersing, flushing, or spraying (1 inch nozzle) in demineralized water at 110°F ($\pm 10^\circ$) for 10 to 15 minutes. Handle with clean rubber gloves at all times during the cleaning operation.

C. Dry - Dry the part by drying in an oven or with missile grade air at 110°F ($\pm 10^\circ$) for 20 to 30 minutes. The part is to be packaged and protected from contamination by wrapping with a minimum of two thicknesses of dry annealed aluminum foil secured with tape.

3.6.1.3 Air lock - All components shall be taken into the final cleaning operation through an air lock.

3.6.1.4 Final cleaning - A Matthews Research bench, or equivalent, with Cambridge absolute filter to filter the incoming air of the bench shall be used in the final cleaning operation. The work bench temperature shall be 72°F ($\pm 5^\circ$) with a maximum relative humidity of 45 percent.

A. Clean nonmetallic components ultrasonically or by using a nylon bristle brush with demineralized water as specified in paragraph 3.2.2.1 or with "Freon," as applicable per paragraph 3.2.1.3.

B. Rinse - Rinse component surfaces thoroughly with prefiltered demineralized water for a period of 5 to 10 minutes.

C. Dry - Dry component in a vacuum oven at 25 to 28 inches of mercury and 100°F ($\pm 20^\circ$).

D. Testing - A Matthews Research bench, or equivalent, employing millipore filter equipment, or equivalent, for particle counts shall be used in the testing operation. (Refer to Paragraph 3.3.1.2.)

E. Dry - Dry component in a preheated 100°F ($\pm 20^\circ$) vacuum oven at 25 to 28 inches of mercury.

F. Assembly and packaging - A Matthews Research Inc. type bench, or equivalent, shall be used for assembly of components.

- Materials and equipment used shall be as specified in 3.3.3 for assembly of components.
- Packaging of assemblies shall be accomplished in this operation by the Saturn Packaging Manual.

G. Air lock - Assemblies after packaging shall leave the assembly and packaging cabinet through an air lock.

3.7 Cleaning Process for Tube Components

3.7.1 Precleaning - Preclean aluminum and stainless steel tubing per MS 100.8. No testing is required at this stage. Drying of components, however, is required. No packaging is required if a delay of no more than 1 or 2 hours is incurred between precleaning and final cleaning operation. The tubes shall be connected to the Whitfield type bench connections before final cleaning operations.

3.7.2 Final cleaning - A Matthews Research bench employing Cambridge absolute filter to filter the incoming air into the work space of the Matthews Research Inc. type work bench, or equivalent, shall be used in the final cleaning operation.

3.7.3 Testing - A Whitfield cabinet type operation employing millipore filter equipment, or equivalent, for particle counts and nonvolatile residue equipment for determining NVR shall be used in the testing operation.

3.7.3.1 Metallic and teflon material shall be free of particles greater than 20 microns in size in any dimension.

3.7.3.2 When testing surface areas of 1 square foot or less, the difference between the used and unused testing solvent shall indicate an increase of not more than 0.0005 g per 100 ml in the nonvolatile residue content of the used solvent over that of the unused solvent.

3.7.3.3 When testing surface areas greater than 1 square foot the increase shall be not more than 0.001 g per square foot. Use "Freon" PCA or alcohol for the solvent in testing.

3.7.4 Nonvolatile Residue (except nonmetallic materials) - The used test solvent, "Freon" PCA or equivalent, contained in the beaker shall be examined for nonvolatile residue as follows:

(a) Evaporate the solvent to 10 to 20 ml volume in a steam bath.

(b) Transfer the solvent to a constant weight (within 0.3 mg) tared 30 ml weighing bottle, weighing to the nearest 0.1 mg.

(c) Continue the evaporation by placing the weighing bottle inside a constant temperature oven which has normalized at 221° to 230° F. Allow weighing bottle to remain inside the oven for a maximum of 1.5 hours or until the solvent is evaporated to dryness.

(d) Remove weighing bottle from the oven and place in a desiccator for cooling.

(e) After cooling remove the weighing bottle from the desiccator and weigh the bottle to the nearest 0.1 mg.

(f) Return the weighing bottle to the normalized, constant temperature oven at 221° to 230° F for 0.5 hour.

(g) Repeat step (d) and step (e). If the difference in the weights taken in step (e) and step (g) is greater than 0.3 mg, repeat steps (f), (d), and (e) until the difference in the weighings is 0.3 mg or less.

(h) Compare the test results obtained from the used solvent to the results obtained from the unused solvent for conformance to the cleanliness level specified herein.

3.7.5 Drying - Dry tubes initially by purging with prefiltered nitrogen or air at 180°F ($\pm 20^\circ$) for 30 to 60 minutes where applicable.

3.7.6 Assembly and Packaging - The parts will be assembled and packaged as specified in the Saturn Packaging Manual.

3.8 Cleaning Process for Spheres

3.8.1 Spheres - These spheres must be cleaned to the cleanliness levels of drawing No. 10419906 for particles and NVR where applicable.

3.8.2 Cleaning Procedure for Spheres - Clean in accordance with M-ME-MPROC-150.8.

4. ACCEPTABILITY PROVISIONS

4.1 The cleaning, processing, and testing of components will meet the 20 micron cleanliness levels on Drawing No. 10419906.

4.2 Other provisions are for particle counts shown in paragraph 3.4.1.4.D.

5. NOTES

5.1 The manufacturing process has been coordinated within the Process Engineering Laboratory and is intended for use in the cleaning, processing, and testing of gas bearing components. The final verification of this procedure will be established during the production of the vehicle.

5.2 Any deviation from this basic procedure will require written concurrence from Process Engineering Laboratory. An emergency concurrence may be made by telephone, but it must be confirmed in writing.

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING PROCESS
FOR CLEANING OF ELECTRICAL CONNECTORS

1. SCOPE

1.1 Scope - This manufacturing process covers the approved Process Engineering Laboratory procedure for the cleaning of electrical connectors which have become dusty and have acquired a slight grease film through open storage.

1.2 Applicability - The requirements set forth in this process are applicable to all standard electrical connectors of a military (MS) or manufacturer's proprietary type, having either hard or resilient (nonmetallic) inserts.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this process, form a part of this process.

SPECIFICATIONS

Federal

O-T-634

Trichloroethylene, Technical, Type II

George C. Marshall Space Flight Center

MSFC-SPEC-237

Solvent - "Freon" Precision Cleaning Agent,
Specification for

MP-6780

Saturn Packaging Procedure Electrical
Connecting Hardware (Proposed)

3. REQUIREMENTS

3.1 Materials

- Polyethylene bags
- Low temperature lubricating grease
"Uni-Temp" Grease, Texas Company, or approved equivalent
- White Petrolatum
Snow White Parmo, Esso Standard Oil Company, or approved equivalent
- Trichloroethylene, Technical, Type II
- "Freon" Solvent - Precision cleaning agent

3.2 Equipment

- Vapor degreaser
- Stainless steel container
- Wire mesh basket

3.3 Procedure

3.3.1 Disassembly

3.3.1.1 Remove all accessories from each connector (i. e., back shell, grommet, O Ring, coupling ring, retaining nut, etc., as applicable.) Do not remove contacts, inserts, or other parts requiring tools to disassemble.

3.3.2 Cleaning

3.3.2.1 Metallic components - Vapor degrease as follows:

- (a) Remove excess lubricant, stains, and heavy concentrations of contamination that would prevent uniform vapor degreasing by wiping with a cloth or paper towel.
- (b) Place components in the vapor degreaser basket in such a manner that complete drainage of the condensed vapors will occur.

- (c) Lower the basket into trichloroethylene vapors which are 188°F ($\pm 3^\circ$) at approximately 11 feet per minute.
- (d) Allow the vapors to condense and flow from the components until the components reach the temperature of the vapor and condensation has ceased.
- (e) Raise the basket to just above the vapors and spray the components with liquid trichloroethylene pumped from the condensate collector.
- (f) If the components are not visually clean, repeat steps (c) through (e) until components are clean.
- (g) Suspend basket of vapor degreased components above the degreaser and allow to dry.
- (h) After components have cooled to room temperature package components by placing in polyethylene bag and heat sealing the bag. Equivalent packaging may be used (covered tote boxes) to prevent recontamination prior to assembly.

3.3.2.2 Nonmetallic and Nonmetallic Combined with Metallic - Clean as follows:

- (a) Place components in a wire mesh basket and place basket in a stainless steel container and fill container with "Freon" until components are covered.
- (b) Agitate the basket for several minutes to remove gross contamination.
- (c) Remove basket of components, pour (or drain) "Freon" from the container, and refill with unused "Freon."
- (d) Submerge basket of components in the unused "Freon" and agitate by raising and lowering the basket for a minimum time of 5 minutes.
- (e) Remove basket of components from container, allow components to drip and "Freon" to evaporate.
- (f) Package components by placing in polyethylene bag and heat sealing the bag. Equivalent packaging may be used (covered tote boxes) to prevent recontamination before assembly.

3.3.3 Assembly

3.3.3.1 Before assembly the accessories should be lubricated as follows:

- For threaded components (coupling rings, retaining nuts, etc.), use a low temperature lubricating grease ("Uni-Temp" Grease - Texas Company), or equivalent.
- For rubber or resilient compounds (O' Rings, gaskets, tapered sleeves, glands, etc.), use white petrolatum (Snow White Parmo-Esso Standard Oil Company), or equivalent.

3.3.3.2 Assemble the cleaned, lubricated connector being careful not to contaminate contacts, solder cups, or either face of the insert with any lubricating grease. Use white gloves.

3.4 Packaging requirements

3.4.1 Skin package and mark each clean, assembled, and inspected connector to corrugated fiberboard as described in detail in procedure MP-6780 and applicable supporting specifications of the Saturn Packaging Manual.

4. ACCEPTABILITY PROVISIONS

4.1 After assembly, and before packaging, each connector is to be inspected for space vehicle use with particular attention to damage that might have occurred during the disassembly, cleaning process, handling, or assembly such as: residual cleaning solution, residue, excess lubrication, softening of insert, bent pins, deterioration of plating, etc. Pin diameters, short pins, and other factors which have previously been inspected and accepted, and which could not have been altered during disassembly, cleaning, handling, or assembly need not be reinspected.

5. NOTES

5.1 This manufacturing process has been coordinated within the Process Engineering Laboratory and is intended for use in the cleaning of electrical connectors for space vehicle use.

5.2 Any deviation from this basic procedure will require written concurrence from the Process Engineering Laboratory. Emergency deviations may be obtained by telephone, but must be confirmed in writing.

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING PROCESS
FOR CLEANING AND TESTING OF
ONBOARD HYDRAULIC SYSTEM COMPONENTS

1. SCOPE

1.1 Scope - This manufacturing process covers the approved Process Engineering Laboratory procedures for the cleaning and testing of on-board hydraulic system components for the S-V vehicle.

1.2 Applicability - The cleaning and testing of all components shall be accomplished by the procedures outlined in this document.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this manufacturing process, form a part of this process to the extent indicated herein.

SPECIFICATIONS

George C. Marshall Space Flight Center

MSFC-PROC-166	Cleaning, Testing and Handling of On-board Hydraulic System Components and MIL-H-5605 Hydraulic Fluid, Procedure for
MSFC-SPEC-217	Trichloroethylene, Technical, Specification for
MSFC-SPEC-237	"Freon" Precision Cleaning Agent
MS 150.0	Manufacturing Specification for Vapor Degreasing of Metallic Surfaces for Space Launch Vehicle Applications

2.2 Other Documents - The following documents form a part of this manufacturing process and unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

Federal

O-S-642	Sodium, Phosphate, Tribasic, Technical, Anhydrous, Dodecahydrate, and Mono-hydrate
O-T-634	Trichloroethylene
O-T-620	Ethyl Alcohol

3. REQUIREMENTS

3.1 Work Area - Components shall be cleaned in the components cleaning room.

3.1.1 Air Conditioning

3.1.1.1 Temperature limits - 72°F (±5°).

3.1.1.2 Humidity limits - The relative humidity will be 45 percent maximum.

3.2 Tagging Components

3.2.1 Upon completion of cleaning, testing, and packaging, all components shall be tagged with the following information.

3.2.1.1 This item has been cleaned and is acceptable for hydraulic system installation. Do not remove seals unnecessarily.

3.2.1.2 Component name and part number.

3.2.1.3 Date cleaned.

3.2.1.4 Inspection stamp.

3.3 Particle size limitations - Upon completion of cleaning and testing, all components and parts shall contain no more than the number of particles shown in Table 5-2.

3.3.1 Cleaning and Test Equipment

3.3.1.1 Matthews Work Bench System

3.3.1.2 Vapor Degreaser

3.3.1.3 Ultrasonic Cleaner

3.3.1.4 Saran Wrap

3.3.1.5 Pyrex filter holder, Millipore Catalog Number XX10004700, or equal, which includes:

- Fritted glass base and rubber stopper.
- Holding Clamp.
- 250 ml Pyrex glass funnel.

3.3.1.6 A filter cover for the glass funnel to minimize contamination from the air passing through the funnel during the vacuum filtration process. The optimum cover would incorporate a Millipore filter in the cover device.

3.3.1.7 A 0.45-micron membrane filter, type HA black grid .047 mm diameter Millipore or approved equivalent.

3.3.1.8 Vacuum flasks.

3.3.1.9 Aspirator or vacuum pump, capable of pulling a minimum of 4 inches of mercury absolute.

3.3.1.10 Plastic petri dishes, disposable, Millipore Catalog PD 10 047 00, or an approved equivalent.

3.3.1.11 Forceps with unserrated tips.

3.3.1.12 Sample bottles, small mouth, glass, etched or otherwise permanently marked to indicate 500 ml sample size.

TABLE 5-2. PARTICLE SIZE LIMITATIONS

Item	Sample Volume	Particle size (microns)				Method
		10-25	25-60	50-100	Over 100 ± fibers	
1. Filter element						
a. As released for space vehicle use	2 liters	50 000	1250	100	10	Millipore
b. Blank analysis (equipment)	2 liters	200	75	15	5	Millipore
2. Components (detail parts) before assembly solvent flushed						
a. Fittings, stainless steel tubing, flexible hose with teflon inner liner.		5	5	0	0	Whatman
b. Bleed valves, check valves, filter housings, manifolds, and quick disconnects.		5	5	0	0	Whatman

NOTES: 1. Uncorrected count - control count not subtracted from test count.

2. These quantities assume normal type and distribution of contaminants. Samples containing excessive proportions of abrasives, metal chips, etc., may be rejected even though the total number of particles is within the prescribed limits.

3.3.1.13 Microscope with mechanical stage capable of magnifications of approximately 45X and 90X.

3.3.1.14 Measuring eyepiece; ocular micrometer.

3.3.1.15 Microscope lamp, high intensity variable 5000 to 6000 candle-power at filter surface and focusing illuminating lens.

3.3.1.16 Wash bottles, polyethylene.

3.3.1.17 Drying and preservation gases - Gases used in drying and preservation processes shall be checked for moisture content and particulate contamination. The dewpoint shall be -65°F maximum. Particle size shall not exceed 20 microns in any dimension.

3.4 Cleaning Processes

3.4.1 Process I, Filters - Filters will be cleaned and tested using the Bendix Sonic Filter Cleaning Console with vacuum attachment.

3.4.1.1 Automatic Operation - Filter elements that can be back flushed will be cleaned by this method.

- Connect filter in position.
- Circulate detergent for 20 to 30 minutes.
- Ultrasonic unit will be activated during this period.
- Intermittent vacuum will be applied during this period.
- Drain tank and back flush with filtered warm water (90 to 110°F).
- Final rinse for 10 minutes with effluent water to drain.
- Dry the filter using hot (160 to 180°F), filtered, missile grade air.

3.4.1.2 Tests

- A. Perform pressure drop test on element as outlined in instruction guide for filter cleaning console before and after cleaning filter elements.
- B. Bubble Testing Filter Element - To determine the maximum pore size of the filter element under test, connect the filter to the outlet provided in the bubble test chamber and proceed as follows:
- Immerse filter element in trichloroethylene, keeping the filter element as close as possible to the surface of the trichloroethylene.
 - Slowly apply air pressure and rotate filter element 360 deg in the trichloroethylene for each change in air pressure.
 - Increase the air pressure until the first bubble appears on the surface of the filter element. The manometer reading at this point is the bubble point of the filter.
 - Correct inches of water to pore size per equation $238/\Delta P$ (inches of water). Maximum allowable pore size by this method is 17 microns. $\Delta P = \text{Final Pressure} - \text{Initial Pressure}$ and $P = \text{Pressure in inches of water}$.
- C. Drain and dry with drying gas.

3.4.1.3 Packaging - Package all filters as specified in Saturn Packaging Manual.

3.4.1.4 Manual Operations - Filter elements that cannot be back flushed will be cleaned in accordance with the instruction guide for the filter cleaning console.

3.4.2 Process II - Components of bleed valves, check valves, fittings, quick disconnects, relief valves, filter housings (where applicable) and manifolds (where applicable), servo valves, actuators, and pumps shall be subjected to final cleaning and testing in a Matthews work bench.

3.4.2.1 Precleaning - Preclean uncoated, metallic and teflon components below per the following instructions, and dry each of the components as required. No packaging is required if a delay of no more than 1 or 2 hours is incurred between precleaning and final cleaning operations. If more than 2 hours delay is incurred, cover the components in covered plastic containers or polyethylene bags.

- A. Vapor degrease - Vapor degrease the parts by lowering into the trichloroethylene vapors. Leave the parts in the vapors until condensation on the surface ceases.
- B. Liquid degrease - Flush or circulate trichloroethylene, at ambient temperature, over or through the surfaces of the components for 10 to 30 minutes. For items requiring removal of varnish type, organic material from the surface, immerse the part in acetone for 10 to 30 minutes.

NOTE: This operation shall be performed in a well ventilated area which shall be free of any possible source of ignition such as static electricity, electric motors, switches, smoking, and open flame. An exhaust hood should be used when available.

- C. Dry - Dry the parts with drying gas (paragraph 3.3.1.17) at 180°F ($\pm 20^\circ$) or dry in a vacuum oven or a drying oven.
- D. Ultrasonic clean - Place the part in an ultrasonic bath consisting of 5 parts detergent to 1000 of water.
- E. Spray rinse - Spray rinse, flush, or immerse with distilled or demineralized water for 10 to 15 minutes at 90°F ($\pm 20^\circ$), or until all sudsing has ceased.
- F. Dry - Components shall be dried using a gas meeting the requirements of paragraph 3.3.1.17, or in a vacuum oven at 180°F ($\pm 20^\circ$) at 5 to 50 mm of Mercury for 1 hour. Items of Grade 300 series stainless steel which have light corrosion at this point shall be processed as specified in paragraph 3.4.2.1.7. Heavily corroded areas shall be cause for rejection of the part.
- G. Phosphoric acid - Parts made of Grade 300 series stainless steel which demonstrate corrosion after processing through paragraph 3.4.2.1.6 shall be immersed in a dilute 10 to 20 percent solution of inhibited phosphoric acid at ambient temperature in an ultrasonic bath for 5 minutes. Rinse and dry the part thoroughly as specified in paragraphs 3.4.2.1.E and 3.4.2.1.F.

- H. Nonmetallic and anodized surfaces shall be cleaned by brushing the part with a nylon brush in a detergent (5 parts detergent to 1000 parts water) solution for 10 to 15 minutes in lieu of solvent degreasing and ultrasonic cleaning. Dowell F-33 is an acceptable nonionic detergent.
- I. Spray rinse - Spray rinse, flush, or immerse with distilled or demineralized water for 10 to 15 minutes at 90°F ($\pm 20^\circ$), or until all sudsing has ceased.
- J. Final cleaning - A Matthews research bench, or equivalent, with Cambridge absolute filter to filter the incoming air of the bench shall be used in the final cleaning operation. The work bench temperature shall be 72°F ($\pm 5^\circ$) with a maximum relative humidity of 45 percent.
- K. Clean nonmetallic components ultrasonically or with prefiltered "Freon."
- L. Rinse - Rinse component surfaces thoroughly with prefiltered "Freon."
- M. Dry - Dry components thoroughly in a vacuum oven at 25 to 28 inches of mercury and at 100°F ($\pm 20^\circ$).
- N. Particle count - The part being tested shall be rinsed with alcohol to remove the water, then flushed with prefiltered alcohol (10 micron level) and a sample taken for particle count. The millipore method for particle count shall be used.
- O. Drying - Place component parts under a dust proof shroud and dry in oven within Matthews work bench.
- P. Packaging - Parts shall be packaged in accordance with the Saturn Packaging Manual, where applicable.

3.4.3 Process III, Stainless Steel Tubing

3.4.3.1 Precleaning - The following precleaning operation will be performed on stainless steel tubing used in hydraulic systems.

A. Preclean the length of tubing thoroughly by flushing with Type II trichloroethylene at the vapor degreaser.

B. Dry the stainless steel tubing by purging with drying gas.

3.4.3.2 Final cleaning

A. Circulate a solution of 10 percent by weight of trisodium phosphate, conforming to Specification 0-5-642, which has been heated to 180°F ($\pm 10^\circ$) through the tubing for 15 to 20 minutes.

B. Rinse for 10 minutes with demineralized water which has been filtered to a cleanliness level of 20 microns.

C. Passivate the tubing by circulating through the tubing for 15 to 20 minutes at 125°F ($\pm 5^\circ$) the following aqueous solution.

20 \pm 2 percent (by volume) nitric acid

2.0 \pm .5 percent (by weight) sodium dichromate

D. Rinse with deionized water until pH of effluent water is in the range of 6 to 8.

E. Particle count - Test the tubing for particles per paragraph 3.4.2.1.J.

F. Dry - Allow the tubing to air dry in the checkout room.

G. Packaging - Tubing shall be packaged in accordance with the Saturn Packaging Manual where applicable.

3.4.4 Process IV (Flexible Hose, Teflon Lined)

3.4.4.1 Brush the "B" nuts with Type II trichloroethylene using a nylon bristle brush. Flush or spray the "B" nuts and the inside of the hose assembly with Type II trichloroethylene, utilizing the pump on the vapor degreaser.

3.4.4.2 Dry with oil free, prefiltered air or nitrogen at ambient temperature for a minimum of 5 minutes until all of the trichloroethylene has been expelled from the hose assembly.

3.4.4.3 Circulate a 10 ± 2 percent solution of trisodium phosphate at $180^{\circ}\text{F} (\pm 10^{\circ})$ for 15 to 20 minutes.

A. Rinse with deionized water until pH of effluent water is in the range of 6 to 8.

3.4.4.4 Flush with alcohol and test the hose for particles per paragraph

3.4.2.1.J.

3.4.4.5 Dry in accordance with paragraph 3.4.3.2.6.

4. ACCEPTABILITY PROVISIONS

4.1 The components shall meet the cleanliness levels of MSFC-PROC-166.

5. NOTES

5.1 This manufacturing process has been coordinated within the divisions of MSFC and is intended for use in the cleaning, processing, and testing of hydraulic components. The final verification of this procedure will be established during the production of the vehicle.

5.2 Any deviation from this basic procedure will require written concurrence from the Process Engineering Laboratory. An emergency concurrence may be made by telephone, but it must be conformed in writing.

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

CLEANING OF S-IC STAINLESS STEEL LOX SUCTION LINES

1. SCOPE

1.1 Scope - This manufacturing process data covers the approved PE Laboratory requirements for the LOX cleaning of the S-IC stainless steel LOX suction lines.

2. EQUIPMENT AND MATERIALS2.1 Equipment

- Cradle structure
- Handling fixtures
- Freon flush unit with accessory equipment
- Hat or funnel type flanges

2.2 Materials

- Freon--PCA MSFC-SPEC-237
- Teflon gaskets or Aclar film
- Drying or preservation gases
- Missile grade air or nitrogen

3. PROCEDURES

3.1 LOX cleaning of tubing and components over 2 inches in internal diameter.

3.1.1 Positioning - Place the LOX line in a vertical position and flange the openings with funnel type or hat shaped open flanges. Insert a teflon-lined flexible hose and nozzle through the opening in the flange at the top of the

vertical length of tubing. The nozzle and attached flexible hose and fittings shall have a protective covering of teflon and be surrounded by teflon bumpers. The tie rod section of line needs to be at the top after the LOX line is positioned vertically into a cradle structure and strapped in.

3.1.2 Freon, PCA Spray Cleaning - Clean by spraying interior of tubing with prefiltered Freon, PCA at 50 psi (± 10) and ambient temperature. Lower and raise the nozzle through at least 3 top-to-bottom-to-top cycles.

3.1.3 Sampling - Sample at least 500 cc Freon, PCA to determine cleanliness according to MSFC-SPEC-164 for particles and NVR. Continue cleaning if sample does not meet cleanliness requirement. Resample after three more cycles of pressure spraying.

3.1.4 Drying - Thoroughly dry the stainless steel tubing by blowing with missile grade air or nitrogen at 180° F ($\pm 20^\circ$) until no moisture can be visually detected on the surface. When the dewpoint reading is between -30 to -40° F, the line is considered dry.

3.2 Protect and package

3.2.1 Seal all openings and protect all exposed surfaces with a film of Aclar or special teflon gaskets and caps with a LOX clean blind flange as provided by R-ME-T.

4. CONTROL

4.1 The following controls shall be exercised as applicable. Particles of contamination per square foot of component surface area shall not exceed the following limit:

- No particle greater than 2500 microns in any dimension.
- One particle between 700 and 2500 microns.
- Five particles between 175 and 700 microns.

NVR content shall meet the LOX requirements of MSFC-SPEC-164.

MANUFACTURING PROCESS DATA
CLEANING AND POLISHING OPTICAL EXPERIMENT CHAMBER
PROJECT NO. 0722

1. SCOPE

1.1 Scope - This procedure describes the methods for cleaning and polishing the inside of the Optical Experimental Chamber in order to minimize any contamination that may be generated when the equipment is subjected to extreme vacuum conditions of 1×10^{-10} Torr. Under these extreme vacuum conditions there is a possibility that oils may out gas from the surface of the metal interior and condense on optical lenses.

The Optical Experiment Chamber is to be made of hot rolled 304 stainless steel and is to be buffed to a No. 7 Standard Mill Finish which is a high lustre polish.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Skin Mill
- Flexible Shaft Buffers
- Vacuum Oven
- Vapor Degreaser
- Hot Water Rinse Tank
- Disc Sander (Hand)
- Drum Sander (Hand)
- Belt Sander (Hand)

- Autoclave, Large, Bldg. 4707 and Required Tubing
- Sand Blast
- Vacuum Leak Detector

2.2 Materials

2.2.1 Abrasive Materials

- 2.2.1.1 Abrasive Paper (Aluminum Oxide, Various size grit)
- 2.2.1.2 Abrasive Discs (Aluminum Oxide, Various size grit)
- 2.2.1.3 Abrasive Wheels (Aluminum Oxide, Various size grit)
- 2.2.1.4 Silicon Carbide Grit Type III Grade B per MIL-A-21380.

2.2.2 Polishing Materials

- 2.2.2.1 Buffing Wheels
- 2.2.2.2 Buffing Cloths
- 2.2.2.3 Lint Free Rag and/or Paper
- 2.2.2.4 Sherwin-Williams "White Lightning" Polishing Compound.

2.2.3 Protecting Materials

- 2.2.3.1 Aluminum Foil Annealed
- 2.2.3.2 Polyethylene Sheeting
- 2.2.3.3 "Shopcoat" peelable coating
- 2.2.3.4 Protective Tapes (various)

2.2.4 Sealing Materials

- 2.2.4.1 Sealing Tape Zinc Chromate (to seal vacuum)

2.2.5 Chemicals

2.2.5.1 Acetone

2.2.5.2 Methyl Ethyl Ketone (MEK)

2.2.5.3 Trichloroethylene

3. PROCEDURE

3.1 Initial Preparation of the Surface

3.1.1 While material is in the flat condition before forming, remove the mill scale and surface imperfections using mechanical abraders. This operation should be conducted in progressive steps using successively finer aluminum oxide grit sizes until a nominal 32 rm finish has been obtained.

3.1.2 Remove all residual dust with an acetone wipe.

3.1.3 Protect the ground and polished surfaces by applying "Shopcoat", adhesive backed peelable, protective coating paper.

3.2 Forming and Fabrication of Chamber

3.2.1 Roll form the finished sheet after protecting the polished surfaces first with "Shopcoat" paper coating and then with aluminum sheeting, to keep the forming rollers from marring the polished surfaces and/or displacing or tearing the paper protective coating.

3.2.2 Strip the paper coating back from the edge as required to accomplish the weld operation.

3.2.3 Clean the areas adjacent to the edges to be welded, just before welding with acetone, or methylethylketone saturated rags.

3.2.4 Proceed with the fabrication of the chamber including the welding and adding of ports, etc.

3.3 Cleaning of Exterior of Chamber

3.3.1 Button up the chamber, take all necessary precautions to protect the interior, and sand blast all exterior surfaces.

3.4 Polishing Interior Surfaces of Chamber

3.4.1 Rough grind weld beads and weld areas flush with the prefinished interior surfaces.

3.4.2 Progressively polish welded surfaces and flange areas, using progressively finer sized aluminum oxide abrasives.

3.4.3 Remove protective paper coatings from the interior surfaces.

CAUTION: Do not wear shoes inside the chamber. Wear booties without shoes, clean room type coveralls, and lint-free gloves. Avoid dropping tools and heavy objects that might mar or scratch the interior surfaces. Use rubber pad.

3.4.4 Remove all residual dust by vacuuming and a final acetone wipe.

3.4.5 Buff all interior surfaces to a No. 7 Standard Mill Finish which is a high luster finish using final polishing materials.

(Suggestion: Use buffing pads and wheels and cloths with "White Lightning" polishing material made by Sherwin-Williams Paint Company.)

3.5 Cleaning Interior Surfaces After Final Buffing

3.5.1 Wipe all interior surfaces free of buffing compound using lint-free wiping pads moistened with acetone.

3.5.2 Carefully inspect all interior surfaces and rework any areas that are dull or nonreflective by buffing and polishing them to the high luster finish. This operation must be completed to the satisfaction of the Project Engineer.

3.5.3 Degrease one section at a time in the trichloroethylene vapor degreaser.

3.5.4 Immerse in hot (preferably boiling) deionized water for 1 hour.

3.5.5 Handwipe all polished surfaces using lint-free soft clean cloths moistened with acetone.

3.5.6 Button up the section by connecting the bulkheads and domes, closing the port holes and preparing the unit for high vacuum. Transfer it to the autoclave.

3.6 Baking the Evacuated Chamber

3.6.1 Insert the chamber into the autoclave.

3.6.2 Connect the evacuation system to the chamber.

3.6.3 Check for leaks during evacuation.

3.6.4 Seal all leaks.

3.6.5 Evacuate the chamber as much as possible. The objective is 12.5 Torr (12.5 mm Hg) or lower.

3.6.6 While maintaining the vacuum, heat the chamber to 600°F. Hold at that temperature for one (1) hour.

3.6.7 Allow the autoclave to cool and, gradually and carefully, break the vacuum.

3.6.8 When conditions permit, enter chamber and inspect it carefully. Remove all residue by wiping with a lint-free, clean cloth moistened with acetone and dry it carefully with a clean, dry cloth. Spot test surfaces by wiping with clean, white, filter paper.

3.6.9 Repeat the steps 3.6.3 through 3.6.8 until no visible residue is found.

3.6.10 When all the visible residue has been baked out of the polished metal interior, seal the cleaned chamber with annealed aluminum foil, taped in place, and cover with waterproof plastic sheeting material in order to protect and preserve the cleanliness of the interior of the chamber.

4. CONTROLS

4.1 Safety. Observe all fire precautions and provide adequate ventilation when handling acetone, methylethylketone, or other similar solvents. Provide adequate ventilation when handling trichloroethylene and other nonflammable

organic solvents. Highly evacuated large chambers and auxiliary systems present implosion hazards.

4.2 Use standard methods for connecting vacuum gauges, manometers, thermometers, and thermocouples.

MANUFACTURING PROCESS DATA FOR CLEANING OF FILTER ELEMENTS

1. SCOPE

This procedure described the method for cleaning and accepting filter elements. The subject elements can range in size from 1/2" O.D. x 1" long to 6" O.D x 20" long. For the purpose of this procedure, a filter is considered to be a cleaning device that has a pore size between 5 microns and 100 microns. The operations covered by this procedure are: visual inspections, bubble point tests, precleaning console backflush cleaning, forward flushing, downstream contamination evaluation, check for non-volatile residue, and packaging.

Filters that are used for many different purposes can be cleaned by this procedure. The various filters may require quite different standards of cleanliness because of these different purposes. The different systems in which filters are used, the specifications that detail the cleanliness requirements, and the operations required for the filters of the different systems are tabulated in Table 5-3.

In addition to the specific types of filter systems that are tabulated, it is possible that there will be miscellaneous filters that do not fall into any of these standard categories. Whenever such filters are cleaned, it is necessary for the organization requesting the cleaning to stipulate the cleanliness level required.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Air pressure regulator for bubble test.
- Wall type manometer.
- Alcohol tray.
- Filtered air supply.
- Scrub brushes, nylon and metal bristles.

TABLE 5-3. PROCESS REQUIREMENTS CLEANING OF FILTER ELEMENTS

System	Cleaning Specification	Visual Inspect Prior to Cleaning	Bubble Test Prior** to Cleaning	Pre-Cleaning	Clean on Console	Forward Flushing	Downstream Part Under Ultrasonic	Particle Check by Hand Flushing	Visual Inspect After Cleaning	NRV Check	Bubble Test** After Cleaning	Package and Ship
LOX	MSFC-SPEC-164	Yes	Yes	Yes	Yes	No	No	Yes	Yes	Yes	Yes	Yes
Fuel	MSFC-SPEC-164	Yes	Yes	Yes	Yes	No	No	Yes	Yes	No	Yes	Yes
Pneumatic on board ground	MSFC-SPEC-164	Yes	Yes	Yes	Yes	No	No	Yes	Yes	No	Yes	Yes
	MSFC-SPEC-164	Yes	Yes	Yes	Yes	No	No	Yes	Yes	Yes	Yes	Yes
Gas Bearing	10419906	Yes	Yes	Yes	Yes	Yes	Yes	No	Yes	Yes	Yes	Yes
Slosh Measuring	10419906	Yes	Yes	Yes	Yes	Yes	Yes	No	Yes	Yes	Yes	Yes
Hydraulic	MSFC-PROC-166D	Yes	Yes	Yes	Yes	Yes	Yes	No	Yes	No	Yes	Yes
Miscellaneous	--	Yes	--	--	--	--	--	--	Yes	--	--	Yes

* Based on Customer Requirements for each filter.

** Bubble test requirement based on the definition that a filter is a particle removing device that has a pore size of less than 100 microns. If such a device has a pore size above 100 microns, then it is no longer considered a filter for the purpose of this procedure.

- Vapor degreaser.
- Beakers, assorted, 500 ml to 1000 ml.
- Bendix cleaning console.
- Adapters for connecting filters to tubing. Should be teflon, gum, rubber, or metal. Should not be black rubber.
- Tubing, I.D. from 3/8 inch to 1/2 inch. Should be teflon, stainless steel, or glass. Do not use tygon.
- Sonic generator transducer and suitable tank.
- Graduated vacuum flask, 2500 ml minimum capacity.
- Vacuum source with vacuum regulator.
- Forced with unserrated tips.
- High pressure membrane filter holders (2 each required)
- Filtration flask with vacuum connection.
- 250 ml Pyrex glass funnel, (filtration).
- Holding clamp for funnel.
- Plastic petri dishes, (disposable).
- Microscope, approximately 45 X and 125 X.
- Plastic bag heat sealer.
- Storage tanks for test fluid (2 each required).

2.2

Materials

- Trichloroethylene, technical, per Specification Q-T-634. This is an appropriate cleaning or test fluid.
- Trifluorotrchloroethylene (Freon) b.p. 47.6°C. This is an appropriate cleaning or test fluid.

- Aluminum foil, household type, approximately 0.0015" thick.
- Tapes, per Specification PPP-T-60.
- Membrane Filter, 0.45 microns, 47 mm diameter.
- Sodium hydroxide, technical grade.
- Nitric acid, technical grade, per Specification Q-A-88.
- Detergent, Bendix 25-I, nonfoaming.
- Ethyl alcohol per Specification Q-E-760.
- Saran wrap, household grade.
- Tape, masking.
- Plastic bags, assorted sizes.
- Labels for filters.

3. PROCEDURES

3.1 Visual Inspection of Uncleaned Filters

3.1.1 Remove the filter from its container (if packaged) or carefully disassemble the filter housing (if applicable). Inspect the filter for breaks, cracks, nicks, dents, or other physical damage. If excessive damage is evident, the filter may be rejected and there is no need to continue with the cleaning operation.

3.1.2 Determine how much precleaning is required on the filter. The amount of precleaning will depend on the level of contamination on the filter.

3.2 Bubble Test Prior to Cleaning Filters

Bubble test all filters before cleaning them. This test will establish the size of the largest pore in the filter. If the pore is too large, the filter may no longer be serviceable and there may not be a need to continue with the cleaning operation.

3.2.1 Preparation of equipment

3.2.1.1 All equipment used in the test setup shall be cleaned prior to assembly. Cleaning shall include solvent degreasing, detergent and water wash, water rinse, and alcohol rinse.

3.2.1.2 Connect test equipment and the filter as shown in Figure 5-2. Fill the container with ethyl alcohol.

3.2.2 Test procedure

3.2.2.1 Holding the filter element in a horizontal position, submerge in alcohol, positioning the element so that the uppermost portion of the element surface is approximately 1/4 inch below the surface of the alcohol.

3.2.2.2 Utilizing the air supply, open the pressure regulator, and slowly pressurize while rotating the element 360°.

3.2.2.3 Increase the pressure until the first bubble appears on the filter element surface.

NOTE: While holding this pressure, bubbles should continue to appear and rise to the surface.

3.2.2.4 Note and record the manometer reading at this pressure. This reading is representative of the largest pore size of the filter element.

3.2.2.5 Regulate the air pressure to 0 psi and allow the alcohol to permeate the element forcing out all air inside the element.

3.2.2.6 Repeat steps 3.2.2.1 through 3.2.2.5 to verify the initial manometer reading.

3.2.2.7 Convert the verified reading to pore size in microns using the following formula:

$$\frac{k}{p} = \text{micron rating of element}$$

Where: p = Manometer reading in inches of water.

k = Conversion constant depending on the make of the filter. This constant will be provided.

1. FILTERED AIR SUPPLY
2. NEEDLE VALVE
3. PRESSURE REGULATOR - 0 to 2 psi
4. FLEXIBLE TUBE CONNECTOR
5. FILTER ELEMENT, SUBMERGED APPROX. 1/4 IN.
6. CONTAINER
7. ETHYL ALCOHOL
8. WALL TYPE MANOMETER

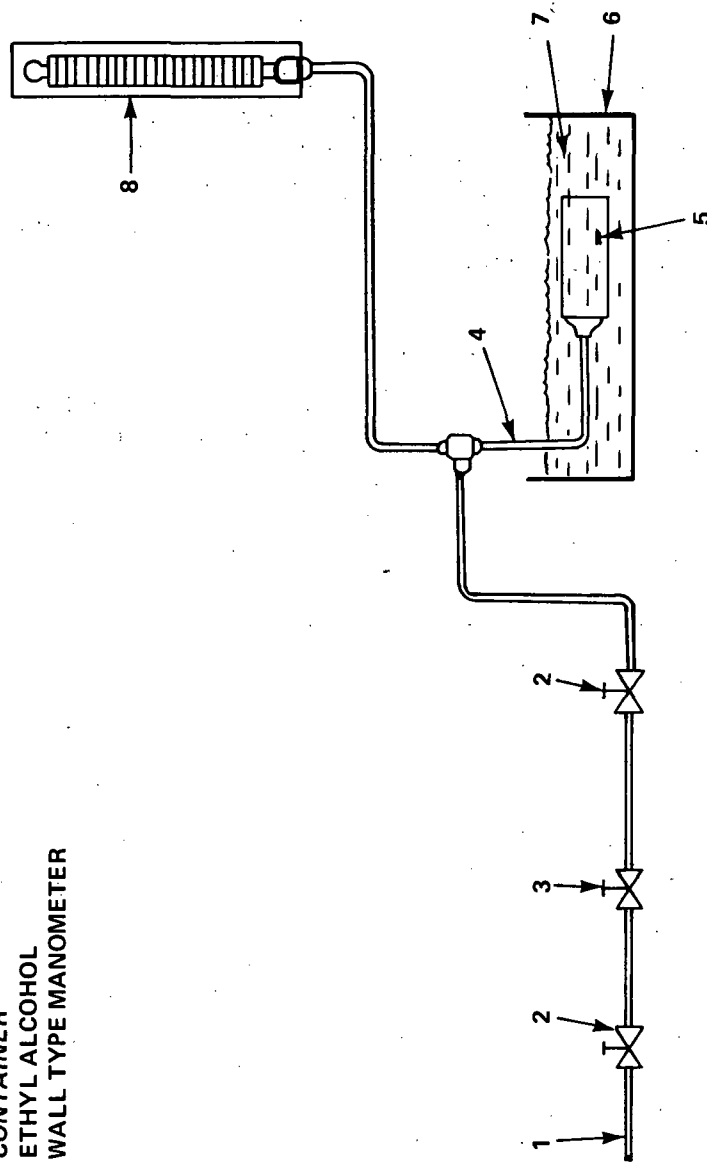


Figure 5-2. Bubble point measuring apparatus.

3.2.2.8 Record maximum pore size reading in microns.

3.2.2.9 Filter elements with maximum pore size results exceeding specified limits of applicable MSFC drawings or documents shall be rejected.

3.3 Precleaning of Filters

3.3.1 Precleaning operations remove the bulk of the contamination from the filter before the final cleaning operation or loosen the contamination so that it can be effectively removed by the final cleaning process. The amount of precleaning needed will depend on the contamination level of the uncleaned filter. Precleaning is divided into two main classes: mechanical cleaning and chemical cleaning.

3.3.2 Mechanical cleaning

3.3.2.1 Brushing - Use brushes to remove contamination from the filter surfaces. The brush should be small enough to get down into the folds of the metal filter cloth. The bristles should be made of a material that is softer than material of the filter so that the filter cloth will not be scored by the brushing action. The brushes should be metal wire or nylon.

3.3.2.2 Compressed air - Use a stream of compressed air to blow the contamination from the filter. Care should be taken not to use excessive air pressure against the filter cloth because the pressure might permanently distort the shape of it.

3.3.3 Chemical cleaning

3.3.3.1 Vapor degreasing - Degrease all filters to remove any oil that might be present.

3.3.3.2 Strong soak (for stainless steel filters only) - If the uncleaned filter element is heavily loaded with contamination, a strong chemical pre-cleaning may be required to loosen the material. The following precleaning soak will normally be adequate.

A. Immerse in hot (180°F) sodium or potassium hydroxide solution (20 percent by weight) for a period of approximately 30 minutes.

B. Rinse in water for 5 minutes.

C. Immerse in 5 percent HNO_3 (Nitric Acid) solution for approximately 10 minutes with frequent agitation of the filter element to promote solution flow through the element pores.

D. Rinse for 15 to 30 minutes in deionized water (pH of 6 to 8) until the pH of the filter is the same as the water supply.

3.3.3.3 Milk soak - If the uncleaned filter is not extremely dirty, a presoak in trichloroethylene or detergent solution may sufficiently loosen the soil for removal.

A. Trichloroethylene soak:

- Soak the filter in trichloroethylene for 30 minutes to 24 hours. If the soil is not loose after 1 day, this method should be abandoned and the hydroxide nitric acid process should be used.

- After the solvent soak, the filter should be dried in a vacuum oven before sonic cleaning.

B. Detergent soak:

- Make up a solution of 6 ounces of Bendix 25-1 nonfoaming detergent to every gallon of water. Heat the solution between 140 to 200°F.

- Soak the filter element in the detergent solution for 30 minutes to 24 hours. If the soil is not loose after 1 day, this method should be abandoned and the hydroxide nitric acid process should be used.

- After detergent solution soak, the filter should be rinsed in water before sonic cleaning.

3.4 Cleaning of Filters on the Bendix Console - The operator should use his judgement in determining the length sonic cleaning time needed for each filter. The filters for gas bearing, sloch measuring, and hydraulic systems require more cleaning on the console than do filters for LOX fuel, and pneumatic systems because of higher cleanliness level requirements.

3.4.1 Instrumentation preparation

3.4.1.1 Close the external electrical disconnect switch located in the line ahead of the cleaner.

3.4.1.2 Open the external valves governing the supply of water and pressurized air to the cleaner.

3.4.1.3 Set the external hot/cold water valves so that the water coming into the chamber is at a minimum temperature of 130 to 140°F.

3.4.1.4 Attach filters to be cleaned to the fittings located on the lid of the cleaning chamber. (A minimum of 8 filters may be cleaned at one time.) Use rubber stoppers or other suitable adapters as aids in connecting the filter to the cleaning chamber fittings.

3.4.1.5 Leave unused ports open.

3.4.1.6 Turn master power switch on. Set the man-auto mode switch to Man. (manual), the timer manual control knob to zero, and the power toggle switch to "On". The warm lamp OPL (white) will then come on.

3.4.1.7 With all drain valves closed, depress the fill button, located on the lower left front panel of the cleaner, to fill the chamber to a depth of 14 inches with 140°F water.

3.4.2 Operation

3.4.2.1 Reseat the lid (with the filters attached) to the top of the unit. Make certain that all hose and electrical connections are secure.

3.4.2.2 After approximately 1 minute of warm up time, the ready lamp AL (amber) will come on. Set the sonics toggle switch to "On". The clean lamp RL2 will come on (red) which indicates that sonic operation has begun in the cleaning chamber.

3.4.2.3 Allow the agitation to continue until the cleaning solution has degassed as indicated by the audible increase in the intensity of sonic hiss coming from the cleaning chamber. This normally takes place within 3 to 5 minutes.

3.4.2.4 After the cleaning solution has degassed, set the sonic switch to off and set the man-auto mode switch to "Auto". Check that the TI timer manual control knob is set at zero and depress the start button (green) and set the sonic switch back on. The system "on" lamp RL1 (red) will light up.

3.4.2.5 Add Bendix 25-I nonfoaming detergent to the cleaning chamber solution at the rate of 3 ounces to every gallon of water. This is approximately 1 pound of detergent powder per fill.

3.4.2.6 Clean the filters for the full 28 minute cycle which includes:

- 12 minutes of sonic cleaning at atmospheric pressure.
- 2 minutes of back flushing to remove dislodged particles of contaminants with sonics on.
- 4 minutes of sonic cleaning.
- 1 minute for automatic drainage of the inner chamber.
- 2 minutes of automatic refilling of chamber with tap water.
- 2 minutes of sonic cleaning at atmospheric pressure.
- 4 minutes to allow container to drain.
- 3 minutes rinse with hot water.
- 7 minutes of drying with electrically heated, filtered compressed air.

3.4.2.7 Sonic cleaning time may be lengthened or shortened by manually selecting any of the automatic operations. The filters that must meet the more stringent requirements will have to be cleaned longer.

3.4.2.8 At the end of the cleaning cycle, lift the lid from the cleaning chamber, and remove the filters.

3.5 Forward Flushing of Filters

3.5.1 General - The filters for gas bearing, slosh measuring, and hydraulic systems require forward flushing because of their high cleanliness requirements. The filters for LOX, fuel, and pneumatic systems do not require it.

Cleaning the filters in the cleaning console only back flushes them. While it might appear that back flushing should completely remove all contaminations that have built up on the filter during use, experience has shown that this is not the case. Many filters that have had extensive back flushing have not passed the cleanliness check, but these same filters have passed the check after forward flushing.

3.5.2 Connect the filter in a flushing system as shown in Figure 2 on Page 10. This system may be altered appreciably to achieve the same result, but it is critical that the filter be positioned upside down and that the circulating solvent is cleaned by passing it through a membrane filter.

3.5.3 With the ultrasonic transducer operating, circulate the solvent through the filter for approximately 30 minutes. This time may be greatly increased or reduced, depending on the type of filter and the degree that it is contaminated.

NOTE: The same setup for forward flushing can be used to conduct the downstream particle check under ultrasonic conditions.

3.6 Inspection of Cleaned Filters

3.6.1 General - Some final inspections are required on all filters that are cleaned, but the type of inspections will depend on the type of filter.

3.6.2 Downstream particle check under ultrasonics

3.6.2.1 General - Filters for gas bearing, slosh measuring, and hydraulic systems require this check. Filters for LOX, fuel, and pneumatic systems do not require it. The same apparatus setup used for forward flushing filters can be used in this check (see Figure 5-3).

3.6.2.2 Procedure for blank

A. General - The test solvent is first checked for cleanliness by passing a portion of it through a clean membrane filter. This is necessary to establish a basis for determining how much contamination is picked up by the test solvent when it is passed through the filter element.

B. Flush membrane filter disc with a minimum of 20 ml test fluid from apparatus (filtered test fluid reservoir) and install in membrane filter holder downstream of the filter element.

C. Pass 2000 ml of test fluid through the apparatus into the graduated vacuum flask.

D. Remove the membrane filter from the filter holder downstream of the adapter using forceps, transfer to a petri dish, cover and mark the petri dish.

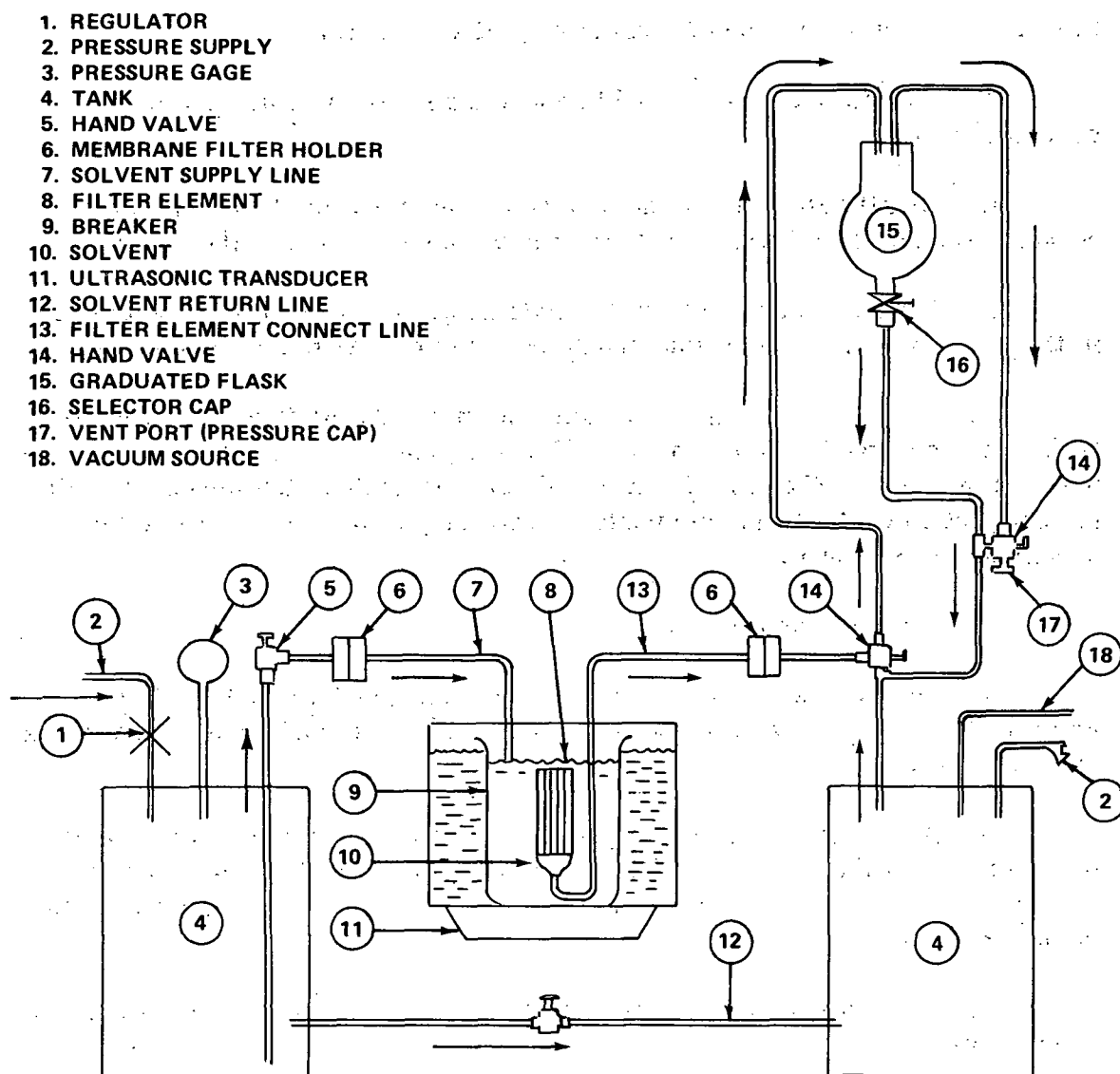


Figure 5-3. Filter flushing system.

E. Determine all particles larger than 10 microns on the entire effective filtering area of the membrane disc. (The total number of particles on the surface can be determined from particle counts made on selected areas of the membrane surface).

F. The contamination count of the blank should not exceed the limits given in Table 5-4.

G. Should the blank value be high, the setup of paragraph 3.6.2.2.3 should be flushed until the count obtained in paragraph 3.6.2.2. F is acceptable.

3.6.2.3 Determination of level of cleanliness of filter

A. The filter element should be handled so as to avoid extraneous contamination.

B. Install filter on a suitable adapter and place the filter into the beaker. Do not allow level of test fluid to go below the top of the filter.

C. Turn on sonic field within 1 minute after immersing of the element in the test fluid. The sonic field should be tuned to maximum intensity.

D. Wait 1 minute, apply vacuum and draw 500 ml of fluid through the filter being analyzed. Repeat at the end of 4 minutes, 7 minutes, and approximately 9 minutes. The last 500 ml should be terminated simultaneously with the completion of the 10 minute period. The temperature of the test fluid should not exceed 90°F.

E. Turn off sonic field.

F. Remove the membrane filter disc from the filter holder downstream of the adapter using forceps, transfer to a petri dish, cover and mark the petri dish.

G. Determine particles larger than 10 microns on the entire effective filtering area of the membrane disc. (The total number of particles on the surface can be determined from particle counts made on selected areas of the membrane surface).

H. Particles of contamination on the filter should not exceed the limits given in Table 5-4.

TABLE 5-4. PARTICLE SIZE LIMITATIONS

Item	Sample Volume	Particle Size (Microns)			
		10-25	25-50	50-100	Over 100 and fibers
1. Blank analysis on test fluid	2 liters	200	75	15	5
2. Analysis on cleaned filter element	2 liters	50 000	4000	100	10

3.6.3 Particle check by hand flushing

3.6.3.1 General - The particle checks for the filters used in the LOX, fuel, and pneumatic systems are made by hand flushing solvent through the surface rather than by drawing the solvent through the filter pores under ultrasonic conditions. Filters for gas bearing, slosh measuring, and hydraulic systems are not checked by the hand flush method.

3.6.3.2 Use a 500 ml (nominal) test solution to determine the particle population left on the filter surface after cleaning. The solution shall be poured over the filter surface in a manner that will trap the solvent after it runs off of the filter.

3.6.3.3 After completion of the rinsing process, the solvent must pass through a membrane filter. The filtered solvent shall be caught in a clean, degreased beaker so that it can be used for the nonvolatile residue check.

3.6.3.4 Determine particles larger than 175 microns on the entire effective filtering area of the membrane disc. The particles of contamination per square foot of filter surface area shall not exceed the following limits:

- No particle greater than 2500 microns in any dimension.
- One particle between 700 and 2500 microns.
- Five particles between 175 and 700 microns.

3.6.4 Visual inspection of cleaned filters

3.6.4.1 Inspect all filters for visible contamination. If such contamination is evident, recleaning may be necessary.

3.6.4.2 Inspect all filters for breaks, cracks, nicks, or dents, or other physical damage. If excessive damage is evident, the filter may have to be rejected.

3.6.5 Check for nonvolatile residue

3.6.5.1 General - Filters for LOX, pneumatic ground support, gas bearing, and slosh measuring systems require a sample to be taken for a nonvolatile residue check. Filters for fuel, pneumatic onboard, and hydraulic systems do not require a nonvolatile residue sample.

3.6.5.2 If the filters have had a particle check by hand flushing, as described in paragraphs 3.6.3.2 and 3.6.3.3, the sample for the nonvolatile

residue check will be available as a by-product of this particle check. If no nonvolatile sample is available, obtain one by submerging the filter into 500 ml (nominal) of test solvent and mildly agitating it. Filter the test fluid through a membrane filter before submitting the sample for the nonvolatile residue check.

3.6.6 Bubble test of cleaned filters - Bubble test all filters after cleaning them. Conduct this test as described in paragraphs 3.2.1 and 3.2.2. This test will establish the size of the largest pore in the filter. If the pore is too large, the filter may no longer be serviceable.

3.6.7 Package and ship

3.6.7.1 Labels for cleaned filters - Make a label for all filters that are cleaned. The label will contain the following information:

- The wording "do not open until ready for use."
- The wording "filter element."
- The part number.
- The serial number.
- Specification to which a part is cleaned.
- The type of system in which the filter will be used.
- Date of cleaning.
- The largest pore of the cleaned filter as determined by the bubble point check.
- The cleaning organization.

3.6.7.2 Packaging - There are two methods for packaging filters. The method used for a filter will depend on the system in which the filter will be used.

A. Packaging of filters for use in hydraulic, gas bearing, and slosh measuring systems - These will be packaged by the following procedures:

- Visually ascertain that filter and packaging material are thoroughly dry before packaging.
- Wrap the filter in abundant layers of suitably cleaned saran wrap or approved equal.
- Overwrap with aluminum foil and secure with tape.
- Overpack with any appropriate packaging material as necessary to prevent damage during storage and handling.
- Place wrapped filter in clean polyethylene bag, press the bag to remove most of the air, and seal with heat sealer.
- Repeat preceding step so that wrapped element is now enclosed in two separate polyethylene bags.
- Attach the label to the filter.

NOTE: The label may be placed between the first and second polyethylene bag.

B. Packaging of filter for use in LOX, fuel, and pneumatic systems - These will be packaged by the following procedure:

- Visually ascertain that filter and packaging material are thoroughly dry before packaging.
- Wrap the filter in a minimum of two layers of aluminum foil. Secure and reinforce with tape.
- Overpack with any appropriate packaging material as necessary to prevent damage during storage and handling.
- Place wrapped filter in clean polyethylene bag, press the bag to remove most of the air, and seal with heat sealer.
- Repeat preceding step so that wrapped element is now enclosed in two separate polyethylene bags.
- Attach the label for the filter.

NOTE: The label may be placed between the first and second polyethylene bag.

3.6.7.3 Shipping - Ship the package filters per the instructions given on the planning work order.

MANUFACTURING PROCESS DATA FOR
CLEANING ATM BLACK BOXES, WITHOUT LENSES

1. SCOPE

1.1 This manufacturing Process Data describes the specific cleaning procedures to be used in cleaning Black Boxes without lenses, and replaces MPD 26003.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

2.1.1 Vacuum Cleaner System and Attachments.

2.1.2 Impulse Heat Sealer.

2.2 Materials

2.2.1 Wiping Cloth, Lint-Free

2.2.2 Freon, Precision Cleaning Agent; MSFC-SPEC-237A

2.2.3 Clean Anti-static Nylon 6 Film for Packaging.

2.2.4 Polyethylene Film for packaging.

3. PROCEDURE

CAUTION: Black Boxes are to be handled with extreme care at all times to prevent damage, such as nicks and scratches.

3.1 Inside the clean room, and wearing clean gloves, carefully remove the protective film bag from the instrument. Avoid generating contamination.

3.2 Carefully vacuum clean all external surfaces. Use hand cleaners with plastic protective nozzles to prevent scratching the surfaces. Clean until there is no visible contamination on the surface of the instrument when it is inspected under a white and black light (ultraviolet light) in accordance with paragraph 3.4.2 Dwg. 50M02412.

3.3 If necessary, wipe the external surfaces clean with lint-free cloths lightly moistened with Freon PCA. Prevent cleaning solution from entering the instrument box. A soft nylon brush may be used to remove adhering particles.

3.4 When the instrument is free of visible contamination and inspected under an ultraviolet light, package it in a clean anit-static nylon inner bag and polyethylene outer bag, and replace it in its original carrying container, if possible.

MANUFACTURING PROCESS DATA PROCESS FOR
CLEANING, PRIMING, FOAM INSULATION AND
SEALING 70" (SHORTENED REDSTONE) CRYOGENIC TEST TANK

1. SCOPE

The procedures set forth in this document will be followed in cleaning, priming, and insulating a 70 inch diameter cryogenic test tank, to be used in evaluating the effectiveness of spray-form insulation.

2. MATERIALS

- Wet/dry sandpaper, 200-grit
- Methyl Ethyl Ketone
- Tellesolve G (Chlorinated hydrocarbons) Narmco Materials
- Trichloroethylene
- Ortho-phosphoric Acid
- Nitric Acid (Concentrated)
- Hydrofluorosilic acid
- Sulfuric Acid (Conc)
- Sodium Dichromate
- Cab-O-Sil
- G-207 Primer
- Toluene
- Foam Insulation Components - CPR-369-3
- EC-2241 Elastomeric Coating (3-M)
- Mica

3. EQUIPMENT

- Steam Jet Nozzle
- Spray Gun (For Primer)
- Hot Hydraulic Airless Spray Foam Equipment (Binks Mfg. Co.)
- Pressure Pots (2) - (Foam Process Co.)
- Radiant Heat Lamps (2 sets)
- R. H. and Amb. Temp. Recorders

4. PROCEDURES

4.1 Cleaning Tank Cylindrical Section

4.1.1 Removal of Cellular Insulation - Peel off cellular structure, including Mylar inner seal, using knife, scraper, or other suitable tool, exposing urethane film on tank wall.

4.1.2 Removal of Urethane Adhesive - Residual adhesive will be removed by one of three acceptable methods:

4.1.2.1 Steam Clean

- Using live steam and a scraper, progressively peel the urethane film from the tank, directing the steam jet directly at the peel line.
- After removal of all detectable traces of adhesive, solvent wipe with trichloroethylene on gauze pads. Dry surface with dry pads before final application of solvent has evaporated.
- Sand paper clean surface with mechanical sander.
- Wipe sanded surface with Methyl Ethyl Ketone.

4.1.2.2 Chemical Stripper

● Apply Tellesolve G, in past form, to areas where urethane is adhered to tank wall. Apply to thickness of approximately 1/16". Allow to act for 30-40 minutes. Remove residue with spatula or scraper.

- After removal of the bulk of the adhesive and stripper, solvent clean with trichloroethylene. Dry surface with dry pads before final application of solvent has evaporated.

- Sand clean surfaces with mechanical sander.

- Wipe sanded surface with MEK.

4.1.2.3 Sanding

- Sand all surfaces until all traces of adhesive have been removed.

- Wipe surface with MEK.

4.1.3 Acid Etch (Cylindrical Section ONLY)

4.1.3.1 Mix British Etch paste and De-Oxidizer as follows:

British Etch

Ortho-Phosphoric Acid 1514 ml

Nitric Acid (Conc) 378 ml

Hydrofluorosilicic Acid 226 ml

Deionized Water 1476 ml

Thicken to past consistency with Cab-O-Sil

Deoxidizer

Sulfuric Acid (Conc) 588 ml

Sodium Dichromate 378g

Deionized water 2840 ml

Thicken to paste consistency with Cab-O-Sil

4.1.3.2 Apply British Etch Solution to cleaned surfaces with spatula to thickness of 1/16"; remove by scraping off excess with spatula, and rinse with water.

4.1.3.3 Apply De-Oxidizer to etched surface to a thickness of 1/16 inch; allow to act for five minutes; remove excess with spatula and water rinse.
"Check pH factor."

4.1.3.4 Air dry with heat gun and protect surfaces with plastic film.

4.2 Cleaning Domes

4.2.1 Foam Removal - Break out or cut out foam insulation presently on tank domes.

4.2.2 Sand Surface - Remove residue attached to dome surfaces by mechanical and/or hand sanding.

(NOTE: It is obvious that foam removal or sanding can not be carried out completely due to skirt interference. Go as far as possible).

4.3 Primer

4.3.1 Primer Mixing - Mix primer in the following proportions:

G - 207-B - 100 g

G - 207-C - 4 g

Toluene - 126 g

MEK - 54 g

4.3.2 Primer Application - Spray coat all surfaces to be insulated with a thin coat of the above proportioned primer. Allow to air dry for 2 hours, then hand rub using gauze pads, to break glaze.

4.3.3 Protect surfaces with plastic film until ready for insulation application.

4.4 Installation of Tank in Vertical Assembly Tower

4.4.1 Move tank to vertical assembly tower and install in horizontal position adjacent to rotating turntable.

4.5 Bulkhead Insulation

- 4.5.1 Set up airless spray equipment on ground level, adjacent to tank.
- 4.5.2 Set protective enclosure over forward end of tank and attach ventilating duct.
- 4.5.3 Go through following procedures in preparation for spraying:
- 4.5.3.1 Check general condition of equipment, paying particular attention to nuts on piston attachment.
- 4.5.3.2 Check solvent supply (1 gal. min.).
- 4.5.3.3 Check pressure pot contents to assure full supply.
- 4.5.3.4 Check nitrogen supply to assure adequate amount to pressurize system. Adjust pressure on pots to 60/95 psi.
- 4.5.3.5 Clean 43-p airless spray gun, tip, and impingement orifices, and assemble gun; using 3120 nozzle (for corners), 0.026" insert, and 0.035" orifices.
- 4.5.3.6 Connect station air supply to formulator; adjust to lowest operable pressure.
- 4.5.3.7 Set temperature to provide the following:
- Component A - 140°F
 - Component B - 80°F
 - Trace Heater - 140°F
- Allow 20 minutes to stabilize; operate gun for 60 seconds (minimum) to flush lines and check heaters under operating conditions; check heaters and adjust as necessary.
- 4.5.3.8 Check RH and temperature recorders for proper functioning.
- 4.5.4 Spray Forward Bulkhead

Spray-foam restricted area at juncture of bulkhead. Rotate tank (by hand), holding 43-P gun (by hand), so that the bulk of the fan pattern will penetrate into the recess. Rotate tank CCW, depositing foam at 270 degree

position. Continue with this operation until foam has risen to join circumferential ring inside skirt.

4.5.4.1 Change nozzle, replacing with 3140 for spraying dome.

4.5.4.2 Rotate tank CCW, holding gun to impinge fan pattern along a generally radial line on dome surface. Hold gun at 270-degree position. Continue spraying until a minimum of 1.75 inches of foam are applied on all dome surfaces.

4.5.4.3 Shut off and flush gun.

4.5.5 Spray-foam Aft Bulkhead

4.5.5.1 Move protective enclosure to cover aft end of tank. Attach ventilating duct.

4.5.5.2 Apply foam in manner identical to process followed in paragraph 4.5.4, spraying forward bulkhead.

4.5.6 Spray Cylindrical Section

4.5.6.1 Tank Erection - Set tank in vertical position on 70-inch rotating turntable. Place portable enclosure over tank and turntable; connect ventilating duct. Establish base line with dial indicator, i.e., check run-out, etc.

4.5.6.2 Move airless spray equipment to moveable platform; install on extension in East end of South section. Only connections to be broken will be nitrogen pressurization system and air line to formulator cylinder.

4.5.6.3 Repeat procedure followed in paragraph 4.5.3, "Preparation for Spraying", with the following exception:

- Install 3150 nozzle, replacing 3140 nozzle used on bulkhead.

4.5.6.4 Mount gun on adjustable column support for automatic spraying. Position support at lowest station initially.

4.5.6.5 Position moveable platform so that 50-degree spray pattern will impinge on an area starting 8 inches (± 2 ") above internal structural ring station. Stand off from tank 24 (± 6) inches to provide minimum overspray.

4.5.6.6 Spray tank per the following sequence:

- Rotate tank to provide tangential speed 12.5 fpm.
- Spray through complete revolution; repeat as necessary to provide minimum of 1.75 inches foam thickness.
- Move up 3 inches by extending support column, and spray for one complete revolution.
- Measure to insure application of min. 1.75 inches foam thickness.
- Repeat (3) above, measuring thickness at completion of each cycle, until support column is fully extended.
- Lower column support, and raise platform to new level which will position gun 3 inches above previous gun location.
- Repeat these functions until the tank is insulated, terminating at a point 8 inches below station of internal stiffener in forward skirt.

4.5.7 Shut-Down - The following procedure will be adhered to in securing the airless spray equipment after completion of insulation application:

- Remove nozzle, nozzle insert, and mixing orifices; clean in MEK or other suitable solvent.
- Cover central opening (from which nozzle was removed) with thumb; turn on solvent wash valve to clean orifice openings; dry, then pack all openings with petroleum jelly.
- Leave all valves open (except air and nitrogen supply valves).
- Shut off air supply at valve between air pressure regulator and air motor.
- Shut off nitrogen supply, and disconnect nitrogen lines from pressure pots.

4.6. Machining or Sanding Surface

4.6.1 Install and turn on dust collection system.

4.6.2 Mount grinder on platform extension - check for perpendicularity ($\pm 1/32$ inch over cyl. length).

4.6.3 Reference position relative to tank reference points.

4.6.4 Rotate turntable in clockwise direction at a tangential speed of 2 feet/minute.

4.6.5 Set grinder to cut (rough cut - not over $3/4$ inch) on lower extreme of insulation.

CAUTION: Set stops to locate cut depth. Do not operate grinder against insulation with turntable stopped. Feed grinder into and out of cut with turntable rotating. Continue upward in overlapping steps until rough cut has been taken over entire surface.

4.6.6 Finish Cut - Repeat process set forth in Paragraph 4.6.5 providing a final thickness of 1.25 (± 0.25) inches.

4.7 Application of EC-2241 Protective Coating

4.7.1 Primary Layer - Apply five box coats, approximately 1.5 mils per coat, with spray paint gun.

4.7.1.1 Drying Time - Allow to air dry for minimum of 15 minutes before application of each subsequent coat. Final coat to air dry minimum of 2 hours.

4.7.2 Surface Voids - Any detectable voids will be filled with a putty consisting of EC-2241 and Mica, formulated in the following proportions:

EC - 2241 - 60 ± 5 parts

Mica - 40 ± 5 parts

Apply in void with spatula; allow to set for 15 minutes; then scrape off excess with spatula.

4.7.2.1 Drying Time - Allow to air dry for two hours prior to application of final layer.

4.7.3 Final Layer - Apply 5 box coats, approximately 1.5 mils per coat, with spray gun.

4.7.3.1 Drying Time - Allow to air dry for minimum of 15 minutes before application of each subsequent coat. Final coat to dry for a minimum of 24 hours before handling.

4.7.4 Control Sample - An aluminum test panel, approximately one foot by two feet in surface dimension, will be positioned vertically adjacent to the spraying station. After each coat application, in both the primary and final layers, a typical box coat will be applied to this test specimen. After the primary layer has cured, and prior to application of the final five coats, mask off 1/2 of the panel area to allow for incremental coating thickness determination. Total coating thickness should be approximately 15 mils.

MANUFACTURING PROCESS DATA FOR CLEANING ATM OPTO-MECHANICAL ASSEMBLIES

1. SCOPE

This Manufacturing Process Data describes the specific cleaning procedures to be used in cleaning the Opto Mechanical Assemblies (with lens).

2. EQUIPMENT AND MATERIALS

2.1 Equipment

2.1.1 Vacuum Cleaner System and Attachments.

2.1.2 Impulse Heat Sealer

2.2 Materials

2.2.1 Wiping Cloth, Lint-Free

2.2.2 Kodak lens cleaner tissue

2.2.3 Freon, Precision Cleaning Agent; MSFC-SPEC-237A

2.2.4 Ethyl Alcohol (Ethanol), O-E-760V.

2.2.5 Clean Anti-Static Nylon 6 Film for Packaging

2.2.6 Polyethylene Film for Packaging.

3. PROCEDURE

CAUTION: Opto-Mechanical black baxes are to be handled with extreme care at all times to prevent damage and misalignment of lens.

3.1 Inside the clean room, and wearing clean gloves, carefully remove the protective film bag from the instrument. Avoid generating contamination.

3.2 Carefully vacuum clean all external surfaces. Use hand cleaners with plastic protective nozzles to prevent scratching the lens. Clean until there is no visible contamination on the surface of the instrument when it is inspected under a white and black light (ultraviolet light) in accordance with paragraph 3.4.2 Dwg. 50M02412.

3.3 If necessary, wipe the external surfaces clean with lint-free cloths lightly moistened with Freon PCA. Prevent cleaning solution from entering the instrument box or contacting the lens.

3.4 If necessary, clean the lens using Kodak lens cleaner tissue, or equivalent, moistened very lightly with ethyl alcohol. A magnetic or radioactive brush may be used to clean the lens.

3.5 When the instrument is free of visible contamination and inspected under an ultraviolet light, package it in a clean anti-static nylon inner bag and polyethylene outer bag, and replace it in its original carrying container, if possible.

MANUFACTURING PROCESS DATA FOR
CLEANING TEMPERATURE TRANSDUCER, SURFACE TYPE 8
NASA DWG. 50M13235 & 50M13242

1. SCOPE

This Manufacturing Process Data describes the procedures for cleaning the temperature gage to be bonded to various surfaces to measure temperatures. It consists of a small plastic rectangle 3/8 x 3/16 inch with a twisted pair of lead wires (one with white insulation - the other with black insulation) covered with white plastic 48 inches long - teflon insulation.

2. EQUIPMENT & MATERIALS

2.1 Equipment

2.1.1 Impulse heat sealer.

2.2 Materials

2.2.1 Wiping cloths, lint-free

2.2.2 Freon PCA, MSFC-SPEC-237A.

2.2.3 Clean anti-static nylon 6 film for packaging

2.2.4 Polyethylene film for packaging.

3. PROCEDURE

3.1 Unwrap and uncoil the wire-carefully paying particular attention not to break, or put undue stress on the wire at the junction of the transducer. (Several units have broken at this point indicating a weakness.)

3.2 Wear clean nylon gloves for the cleaning and packaging procedures.

3.3 Cleaning procedures - Hand wipe the wire covering and the surface of the attached transducer with lint-free wipes moistened with Freon PCA until they are visibly clean.

3.4 Packaging procedures - Recoil the wire carefully and insert the transducer and the connecting wire into an antistatic Nylon 6 Bag, in such a way that the wire junction is protected, and seal it.

3.5 Tape the nylon bag to a metallic stiffener to prevent stressing of the part.

3.6 Double bag the elements, inserting the identification papers inside the polyethylene bag so that they may be read and seal the outer bag.

SECTION VI. SUPERALLOYS — NICKEL AND COBALT BASED ALLOYS

This section contains the chemical process data developed for the superalloys. Presently materials and new processes are being developed for Space Shuttle Application. This data will be added at a later date when complete.

- Cleaning of Hastelloy "C" tubing before welding
- LOX Cleaning of Hastelloy Tubing and Fittings

CLEANING OF HASTELLOY "C" TUBING BEFORE WELDING

1. SCOPE

1.1 This document applies specifically to the surface preparation of Hastelloy "C" tubing prior to welding.

2. EQUIPMENT AND MATERIALS

2.1 Equipment — Vapor Degreaser and accessories, wire baskets, hoist, soak cleaner tank, pickling tank, rinse tanks, and handling devices.

2.2 Materials — Silicon carbide or aluminum oxide abrasive cloth (280 grit or finer), nylon cord, soak cleaner (Wyandotte Altrex), pickling solution (1 to 1.5 percent hydrofluoric acid and 15 to 20 percent nitric acid), demineralized and tap water, missile grade air, and Aclar film (33C).

3. PROCEDURE

3.1 Mechanically clean the interior and exterior of the tubes for a distance of 1 inch from the ends, using a silicon carbide or aluminum oxide abrasive oxide cloth (280 grit).

3.2 Position the tubing in a vertical position by tying to a wire basket or other handling device with a nylon cord.

3.3 Soak clean the entire tube by immersing in a solution of Wyandotte Altrex.

NOTE: This step is initially required to remove white drawing compound.

3.4 Rinse in agitated tap water at 130° to 150° F for a minimum of 10 minutes.

3.5 Blow out excess water with pressurized air.

3.6 Vapor degrease the entire tubing. Spray the tubing with the spray nozzle.

3.7 Pickle the tube by immersing in a solution containing 1 to 1.5 percent HF and 15 to 20 percent HNO₃ at 95° F (±5 °) for 10 minutes.

LOX CLEANING OF HASTELLOY "C" TUBING AND FITTINGS

1. SCOPE

This manufacturing process data covers the method for LOX cleaning Hastelloy "C" tubing and fittings.

2. EQUIPMENT AND MATERIALS2.1 Equipment:

- Vapor degreaser
- Tube cleaning console
- Millipore filter equipment
- Protective clothing
- Asbestos clothing
- Eye shields
- Handling tongs

2.2 Materials:

- Trichloroethylene O-T-634
- Nitric acid O-N-350
- Aclar film (33C)
- Demineralized water
- Sodium dichromate O-S-595
- Alcohol, ethyl, specially denatured MIL-A-6091
- Drying or preservation gases, MSFC-SPEC-164

3. PROCEDURE

3.1 LOX Cleaning Procedure (Internal Surfaces Only)

3.1.1 Circulate a solution of 10 (± 2.0) percent by weight of trisodium phosphate through the tubing at 180°F ($\pm 10^\circ$) for 10 to 15 minutes.

3.1.2 Circulate demineralized water at a temperature of 160 to 180°F through the tubing for 5 to 10 minutes.

3.1.3 Neutralize with 20 to 30 percent nitric acid by recirculating for 5 to 10 minutes at ambient temperature.

3.1.4 Circulate demineralized water at a temperature of 160 to 180°F through the tubing for 5 to 10 minutes.

3.1.5 Passivate the tubing by circulating through the tubing for 15 to 20 minutes at 125°F ($\pm 5^\circ$) the following aqueous solution:

20 (± 2) percent by weight nitric acid
2.0 (± 0.5) percent by weight sodium dichromate

3.1.6 Circulate demineralized water through the tubing at ambient temperature until the pH of the effluent is 6 to 8.

3.1.7 Using nitrogen or air at 180°F ($\pm 20^\circ$), dry by flushing gas through the interior of the tubing for 15 to 20 minutes at 15 to 25 pounds per square inch pressure.

3.1.8 To determine the cleanliness level, circulate trichloroethylene through the tubing until the particle count does not exceed the following: No particle greater than 2500 microns in any dimension, one particle between 700 and 2500 microns, five particles between 175 and 700 microns. The nonvolatile residue shall be not greater than 0.001 gram per square foot of surface area.

3.1.9 Using nitrogen or air at 180°F ($\pm 20^\circ$), flush gas through the interior of the tubing for 15 to 20 minutes at 15 to 25 pounds psi pressure. The drying gas shall be prefiltered to a 100 micron level (absolute), the oil content shall be no greater than 3 parts per million by weight, and the moisture content shall not be greater than 24 parts per million by volume.

3.1.10 Tubing ends shall be sealed with Aclar film(33 C) to prevent contamination after cleaning. The film shall be secured to the tubing with masking tape.

4. CONTROLS

4.1 Personnel shall insure that the following controls are exercised as applicable:

4.1.1 Safety measures must be exercised to avoid spilling or splattering of the acid solutions. Cautiously raise or lower tubing into the bath. Protective devices such as asbestos gloves and apron, handling tongs, eye shields, etc., are used.

4.1.2 Adequate ventilating hoods shall be employed over acid and alkali tanks to remove injurious fumes from the area.

SECTION VII. CLEANING TITANIUM ALLOYS

This section contains the processes for titanium alloys that were developed and successfully used in the S& E-PE Laboratory.

- Manufacturing Process Data-Cleaning of Titanium Alloy 8A1-Mo-IV.
- Cleaning ATM Components Titanium, Beryllium and Stainless Steel Alloys.

MANUFACTURING PROCESS DATA FOR CLEANING OF TITANIUM ALLOY 8A1-1Mo-1V

1. SCOPE

1.1 This manufacturing process data covers the cleaning procedure for titanium alloy 8A1-1Mo-1V that has not been subjected to heat treating above 1100° F, or has been pretreated before heat treating to reduce scale formation.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Polyethylene graduated cylinder
- Polyethylene or acid resistant cleaning container or tank
- Cold water rinse tank
- Hot water rinse tank
- Exhaust hood or well ventilated area
- Handling tongs or equipment
- Clock or timer
- Tank for Turco 4215

2.2 Materials

- Deionized Water
- Turco 4215 Non-silicated Alkaline Cleaner
- Nitric acid concentrated (approximately 70 percent HNO_3), reagent grade
- Hydrofluoric acid (48 to 52 percent HF)
- Acetone (99.5 percent) ACS Specs. reagent grade

3. PROCEDURES

3.1 Preparation of the acid pickling bath.

NOTE: This pickling bath requires a special tank.

	<u>Percent Weight</u>
3.1.1 Add water deionized	60.0
3.1.2 Add nitric acid conc (70 percent HNO ₃)	35.0
3.1.3 Add hydrofluoric acid (48 to 52 percent HF)	5.0

3.2 Cleaning

3.2.1 Remove all grease or markings by immersing the parts to be cleaned in acetone at ambient temperatures, or wipe parts thoroughly with acetone saturated clean cloths or clean paper wiping materials.

3.2.2 Immerse in a non-silicated alkaline cleaner such as Turco 4215 at about 10 ounces per gallon at 170°F (±5°) for 10 to 15 minutes.

3.2.3 Rinse the parts in deionized water at 95°C (200°F).

3.2.4 Immerse the part in the acid pickling bath for a minimum of 60 seconds. Longer pickling times will depend on the condition of the pretreated titanium.

3.2.5 Rinse the parts thoroughly in deionized water at ambient temperatures.

3.2.6 Air dry at room temperature.

4. CONTROLS

4.1 Observe all the safety precautions necessary for handling hydrofluoric acid.

4.2 The deionized water used should have a specific resistance of not less than 50 000 ohms and a range of pH within 6.0 to 8.0.

4.3 The acid pickling solution should be maintained at a pH no greater than 1.5 or should never drop below 20 percent HNO_3 .

NOTE: The pickling solution contains hydrofluoric acid and requires a specially lined tank.

4.4 No halogenated solvents shall be used.

4.5 Care must be taken to prevent contamination from any chlorides, and other contaminants such as fingerprints, etc.

4.6 Protect the cleaned parts in clean wrapping paper.

CLEANING ATM COMPONENTS
BERYLLIUM, TITANIUM AND STAINLESS STEEL ALLOYS

1. SCOPE

1.1 Scope - This manufacturing procedure covers the final cleaning and packaging of ATM components.

1.2 Applicability - The precleaning and final cleaning are applicable where appropriate.

- Titanium
- Beryllium
- Stainless Steel

2. APPLICABLE DOCUMENTS

SPECIFICATIONS

Federal

Federal Standard 209A Clean Room and Work Station Requirements and Controlled Environments.

George C. Marshall Space Flight Center

MSFC-MPROC - 151 Contamination Control and Environmental Protection of Space Vehicle and Associated Equipment, Procedure for

MSFC-SPEC-164 Cleanliness of Components for use in Oxygen, Fuel and Pneumatic Systems

MSFC-STD-246 Design and Operational Criteria of Controlled Environmental Areas

MSFC 30M14500 Apollo Telescope Mount Assembly

MSFC 50M02412 ATM Cleanliness Specification

3. REQUIREMENTS

3.1 Facility Cleanliness

3.1.1 Environmental cleanliness levels required for cleaning and packaging per 50M02412

- Class 30 000 clean rooms — 30 000 particles per cubic foot greater than 0.5 micron and 215 particles greater than 5 microns for both the spar and rack-mounted optical component.
- Class 100 000 clean rooms — 100 000 particles per cubic foot greater than 0.5 micron and 700 particles per cubic foot greater than 5.0 micron.
- Relative humidity shall not exceed 50 percent at 72° F.

3.2 Equipment

3.2.1 Vacuum Oven

3.2.2 Heat Sealing

3.2.3 Ultrasonic

3.3 Chemical and Materials

3.3.1 Acetone

3.3.2 Ethanol

3.3.3 Tri Fluoro, Trichloro Ethane (Freon)

3.3.4 MEK

3.3.5 Dowell F-33

3.3.6 Polyethylene

3.3.7 Nylon - 6 Package

3.3.8 Trichloroethylene

3.3.9 Nylon Gloves and Cloths

3.3.10 Xylene

3.4 Requirements — No halogenated solvents permitted for beryllium and titanium alloys.

4. CLEANING

4.1 Phase I — Titanium - Rack

4.1.1 Preclean — Parts shall be cleaned with acetone or MEK to remove grease, oil, ink, or dirt.

4.1.2 Parts shall be ultrasonically cleaned with acetone or MEK to remove any residual oil, grease, or ink left from precleaning.

4.1.3 Parts shall be detergent cleaned in an ultrasonic bath at 130° F ($\pm 10^\circ$) for 3 to 5 minutes with Dowell F-33.

4.1.4 Rinse — Parts shall be rinsed in D.I. water until suds free with an additional 5 minute rinse.

4.1.5 Dry — Parts shall be air dried with hot missile grade air at temperature of 150 to 180° F.

4.1.6 Final Cleaning — Parts shall be flushed with acetone or MEK.

4.1.7 Cleanliness — Particle and NVR levels for final cleaning shall be specified by MSFC-SPEC-164.

4.1.8 Dry — Parts shall be dried in vacuum oven at 25 to 27 inches of mercury at temperature of 100 to 110° F until dry.

4.1.9 Package — Parts shall be double-bag sealed with inner bag being nylon-6 and outer bag being polyethylene.

4.2 Phase II — Beryllium - ATM Camera

4.2.1 Preclean — Parts shall be cleaned with a nylon cloth moistened with acetone, MEK, or Xylene to remove any grease, oil, ink, or dirt.

4.2.2 Parts shall be ultrasonically cleaned in acetone, MEK or Xylene.

- 4.2.3 Final cleaning — Parts shall be cleaned in acetone, MEK, or Xylene.
- 4.2.4 Cleanliness — Particle and NVR levels for final cleaning shall be specified by MSFC-SPEC-164.
- 4.2.5 Dry — Parts shall be dried in vacuum oven at 25 to 27 inches of mercury at 110° ($\pm 10^\circ$) until dry.
- 4.2.6 Package — Parts shall be double-bag sealed with the inner bag being nylon 6 and outer bag being polyethylene.
- 4.2.7 Caution — Beryllium is TOXIC - handle with care by using nylon gloves for all cleaning operations.
- 4.3 Phase III — Stainless Steel - ATM Camera.
- 4.3.1 Preclean — Parts shall be cleaned with acetone or MEK to remove grease, oil, ink, or dirt.
- 4.3.2 Parts shall be vapor degreased in trichloroethylene.
- 4.3.3 Parts shall be ultrasonically cleaned with detergent to remove any residual oil, grease, or ink left from precleaning.
- 4.3.4 Rinse — Parts shall be rinsed in D.I. water until suds free with an additional 5 minute rinse.
- 4.3.5 Dry — Parts shall be air dried with hot missile grade air at temperature of 150 to 180° F.
- 4.3.6 Final clean — Parts shall be flushed with acetone or MEK.
- 4.3.7 Cleanliness — (Refer to paragraph 4.1.7)
- 4.3.8 Dry — Parts shall be dried in vacuum oven at 25 to 27 inches of mercury at temperature of 100 to 110° F until dry.
- 4.3.9 Package — Parts shall be double bad sealed with inner bag being nylon - 6 and outer bag being polyethylene.

5. **QUALITY ASSURANCE**

5.1 **Inspection Points**

5.1.1 **Titanium and Beryllium.**

5.1.1.1 Refer to paragraph 4.1.7

5.1.1.2 Refer to paragraph 4.2.4

5.1.2 **Stainless Steel**

5.1.2.1 Refer to paragraph 4.3.6

SECTION VIII. CLEANING CARBON AND MILD STEELS

The methods used at the S&E-PE Laboratory for cleaning mild steel are presented in this section of the manual.

- Manufacturing Specification for the Cleaning of Carbon Steel Alloys
- Manufacturing Process Data - Cleaning of Mild Steel Bottle with Single Opening
- Manufacturing Process Data - Cleaning of Mild Steel Bottle with Multiple Openings
- Manufacturing Process Data - Mild Steel Containers and Tubular Assemblies Chemical Cleaning of

MANUFACTURING SPECIFICATION FOR THE CLEANING OF CARBON STEEL ALLOYS

1. SCOPE

1.1 Scope - This manufacturing specification covers the approved PE. Laboratory manufacturing requirements for cleaning carbon steel prior to the application of surface treatments.

1.2 Applicability - All steels containing less than seven percent of alloying elements (not including metalloids) and a minimum of 90 percent of iron shall be covered by this specification.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this specification, form a part of this specification.

SPECIFICATIONS

Federal

0-A-51C	Acetone, Technical
0-S-809A	Sulphuric Acid, Technical
0-0-670	Orthophosphoric (Phosphoric) Acid, Technical
0-T-634A	Trichloroethylene, Technical
P-C-436A	Cleaning Compound, Alkali Type
TT-C-490	Cleaning Methods and Pretreatment of Ferrous Surfaces and Organic Coatings
P-C-535 (1)	Cleaning Compound, Platers Electro-Cleaning, For Steel

Military

MIL-C-490A	Cleaning and Preparation of Ferrous and Zinc Coated Surfaces for Organic Protective Coatings
MIL-R-8633A (Aer) (2)	Remover, Paint, Nonflammable, Water-Rinsable.
MIL-R-25134 (2)	Remover, Paint and Lacquer, Solvent Type

George C. Marshall Space Flight Center

MS 150.0	Manufacturing Specification for Vapor Degreasing of Metallic Surfaces
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(Copies of specifications, standards, drawings, and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Other Publications - The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on date of issuance of this specification shall apply.

Amchem Products, Inc.

1413D-8-59

Rodine No. 50, Inhibitor

(Copy of this publication may be obtained from Amchem Products, Inc., St. Joseph, Missouri.)

Cleveland Metal Abrasive Company

Catalog 159

G-50 Grit

(Copy of this publication may be obtained from Cleveland Metal Abrasive Company, 888 E. 6th Street, Cleveland, Ohio.)

Microbeads, Inc.

MB-DS-1-1159

The Story of Glass-Shot

(Copies of this publication may be obtained from Microbeads Inc., P. O. Box 241, Jackson, Mississippi.)

Oakite Products, Inc.

Bulletin, 12/58

Oakite Ruststripper

Oakite Service
Bulletin No. 7B

Oakite Stripper S-A

(Copies of this publication may be obtained from Oakite Products., Inc., 19 Rector Street, New York, New York.)

Pangborn Corporation

Bulletin No. 333A

Hydrofinish Abrasive

(Copy of this publication may be obtained from Pangborn Corporation, 10 Pangborn Blvd., Hagerstown, Maryland.)

Turco Products, Inc.

Bulletin No. 74

Turco W. O. No. 1

Bulletin No. 76

Turco Vitro-Klene

Bulletin No. 165

Turco 3878

Bulletin No. 168

Turco 3094

(Copies of these publications may be obtained from Turco Products, Inc., P. O. Box 1055, Wilmington, California.)

Wyandotte Chemical Corporation

Pamphlet F-2780

Wyandotte B. N.

(Copies of this publication may be obtained from Wyandotte Chemical Corporation, Wyandotte, Michigan.)

3. REQUIREMENTS

3.1 General - No deviation from the requirements of this manufacturing specification shall be permitted without prior written approval of the MSFC initiating activity.

3.2 Materials

3.2.1 Acids

3.2.1.1 Sulphuric acid - The sulphuric acid used for descaling or for stripping phosphate coatings shall meet Specification 0-S-809A.

3.2.1.2 Orthophosphoric acid - The acid used for fingerprint removal or in rinsing water prior to phosphating shall meet Specification 0-0-670.

3.2.2 Trichloroethylene - The trichloroethylene used shall conform to Specification O-T-634A.

3.2.3 Hot alkaline degreaser - The compound used for hot, dilute, aqueous degreasing solutions shall be equal or equivalent to Turco Vitro-Klene and shall meet Specification P-C-436A.

3.2.4 Electrocleaner (plater's) - The cleaning compound used in electro-cleaning solutions shall be equal or equivalent to Oakite 90 and shall meet the requirements of Specification P-C-535(1).

3.2.5 Rust remover (fingerprint) - The compound used for the removal of corrosion resulting from fingerprints shall be equal to or equivalent to Turco W. O. No. 1.

3.2.6 Rust remover - The compound used for removing thin layers of rust or oxide shall be equal or equivalent to Oakite Ruststripper.

3.2.7 Abrasive (Hydrohone) - The abrasive used in hydrohoning may be Pangbornite, Hydrofinish, Abrasive (No. 100 or No. 200) or Microbead, Spherical Bead, Abrasive MS-ML, or equivalent.

3.2.8 Metal abrasives - Metallic shot or grit employed to remove heavy oxide or rust shall be equal or equivalent to Cleveland Metal Abrasive Company's normalized product.

3.2.9 Paint remover - The paint remover used for removing epoxy formulated paints shall be equal or equivalent to Turco 3994 or Oakite Stripper S-A.

3.3 General Cleaning Procedures

3.3.1 Precautions

3.3.1.1 General - When assemblies are cleaned, no solution containing an acidic substance shall be used if entrapment of the solution occurs.

3.3.1.2 Masking - Mask all plastics, rubber, fabrics, and bushings.

3.3.1.3 Pickling limitations - Avoid the use of acid solutions to remove heat-treat scale from steels heat treated to an ultimate tensile strength of 220 000 psi and above. Steels heat treated below and ultimate tensile strength of 220 000 psi may be acid cleaned, provided they can be given the hydrogen embrittlement relief treatment outlined in paragraph 3.3.1.4.

3.3.1.4 Relief of hydrogen embrittlement - When corroded steels having a hardness of Rockwell C-scale 35 and not over C-48 are cleaned, they shall be heated at 375° F ($\pm 10^\circ$) for 3 hours prior to any stripping operation (plating or epoxy paint removal). All components shall be given a hydrogen embrittlement relief after any acid treatment according to the following schedule.

A. Rockwell C-scale 35 to 39 - Uncarburized steel parts having a hardness between Rockwell C-35 and C-39 shall be heated at 375° F ($\pm 25^\circ$) for 4 hours.

B. Rockwell C-scale 40 to 47 - Uncarburized steel parts having a hardness between Rockwell 40 to 47 shall be heated for 24 hours at 375° F ($\pm 25^\circ$).

C. Rockwell C-scale 48 and above - Parts having a hardness of Rockwell C-48 and above shall not be subjected to pickling or phosphating procedures.

3.3.1.5 Mechanical cleaning requirements - When corroded or oxidized parts above 42 Rockwell C-scale are cleaned, they must be mechanically or pneumatically descaled per paragraphs 3.3.3.2.C, 3.3.3.2.D and 3.3.3.3.

3.3.2 Degreasing

3.3.2.1 Solvent cleaning - When marking inks or other resistant soils exist, they shall be removed by acetone meeting Specification O-A-51c.

3.3.2.2 Vapor degrease - Parts shall be vapor degreased per MSFC Specification MS 150.0 when vapor degreasing is desired.

3.3.2.3 Primers, paints, plating, and coatings - These shall be removed when it is desired by the methods given in the pertinent paragraphs of this specification.

3.3.2.4 Alkaline cleaning - When parts are not oxidized and a surface free of water-break is desired for a number of surface coatings, they shall be immersed in an aqueous solution of 8 to 10 ounces per gallon of Turco Vitro-Klene or equivalent at a temperature of 195 to 205° F for approximately 20 minutes. Following this immersion they shall be rinsed and dried if desired. Any alkaline cleaner employed must be equal to or exceed the cleaning efficiency of the standard comparison cleaning compound given in Specification P-C-436a.

3.3.2.5 Electrocleaning - Parts may be electrocleaned by the following method for the removal of soils other than oxide. The solution shall have a concentration of 4 to 8 ounces per gallon of water of Wyandotte B. N. or equivalent and operate at a temperature of 180 to 200° F. The part shall be the anode and the voltage imposed shall maintain a current density of 40 to 80 amperes per square foot. To facilitate a rapid removal of soil, it is permissible to periodically reverse the polarity. Following the electrocleaning, parts shall be rinsed thoroughly and dried if desired. The electrocleaning compound used must be equal to or superior to requirements of Specification P-C-535(1).

NOTE: Attention is called to paragraphs 3.3.1.4 and 3.3.1.5

3.3.3 Corrosion removal

3.3.3.1 Fingerprints - Corrosion from fingerprints should be removed by an ethyl alcohol solution of phosphoric acid unless prohibited by engineering drawing or the purchasing agreement. Turco W. O. No. 1 or equivalent in concentrations of one part W. O. No. 1 to three parts water may also be used. Following the application of the acid, the area shall be wiped dry.

3.3.3.2 Thin heat scale or corrosion films - Parts shall be descaled and cleaned by immersing in an aqueous solution of Oakite Ruststripper or equivalent at a concentration of 2 to 3 pounds per gallon and at a temperature of 200° F ($\pm 10^\circ$) for 20 to 30 minutes. Following rust removal, parts shall be thoroughly rinsed in cold flowing water and dried if desired. While this descaling bath will also remove oil and grease, heavy contaminations should be removed per paragraph 3.3.2.1.

NOTE: Do not employ direct current in the use of this alkaline bath. Bath is approved only as a soak-type cleaner.

A. Special rinse No. 1 - If parts are descaled prior to phosphating, a second water rinse containing 2 percent phosphoric acid should be used to meet paragraph 4.2.3 of Specification MIL-C-490A.

B. Special rinse No. 2 - If parts are to be painted, the second rinse water should contain one-tenth of a percent by weight of chromic acid.

C. Mechanical removal of light scale - When preference exists for mechanical removal of light scale, parts shall be hydrohoned using as the abrasive, either Pangbornite Hydrofinish Abrasive (No. 100 or No. 200) or Microbead Spherical Bead Abrasive MS-ML.

D. Mechanical removal of light scale by abrasion - Where dimensional tolerances permit, parts may be hand or power, wire-brushed, rubbed with steel wool, sanded by hand, or power-operated emery cloth.

3.3.3.3 Removal of resistance or heavy scale - The removal of heat scale, casting scale, weld scale, and corrosion shall be accomplished by either pneumatic or mechanical means. The metallic grit or shot and mineral or vitreous abrasive employed shall be 90 percent on 45 mesh when new and not used when it reduces to below 100 mesh.

3.3.3.4 Power tool scale removal - If the tolerances on semi-finished parts permit, the use of power tools for localized grinding is permissible, if it is performed on parts having a hardness less than 40 Rockwell C-scale and subsequently magnafluxed for surface crazing.

NOTE: When scale is impregnated with oil or grease, the part should first be degreased and dried to a practical degree before blasting with grit, shot, or mineral abrasives.

3.3.4 Removal of electrodeposited metals - All electroplated metals shall be stripped per the pertinent manufacturing or process specification. The treatment prior to stripping, the stripping solution, and the treatment shall conform to the requirements of the specification.

3.3.5 Paint removal

3.3.5.1 Brush or spray (stripping) - The paint remover, meeting specification MIL-R-25134 (2) or MIL-R-8633A, shall be applied and allowed to remain in contact with the coating for 5 to 10 minutes to loosen the coating. The surface shall be kept moistened with paint remover and subsequently hosed off with tap water. The part should be dried to lessen the possibility of rust formation.

NOTE: Paint strippers must not contact organics such as rubber, plastic, sealants, and fabrics.

3.3.5.2 Epoxy paint removal - When paints such as epoxy formulations are resistant to the paint removers in paragraph 3.3.5, Turco No. 3994 or Oakite Stripper S-A shall be applied and allowed to remain in contact with the coating until the surface begins to wrinkle. High pressure water is to be used to remove the coating. Light brushing with a bristle brush may assist in the removal of stubbornly adhering paint.

3.3.5.3 Paint removal from parts over 220 000 psi tensile strength - Paint shall be removed by blasting or hydrohoning with Microbead Spherical Bead Abrasive MS-XL or Pangbornite Hydrofinish abrasive.

3.3.6 Removal of phosphate coatings - When phosphate coatings must be removed, the parts may be pickled in a solution of 10 percent by volume of sulfuric acid inhibited with Rodine No. 50 or equal, at room temperature, or immersed in an alkaline bath given in paragraph 3.3.3.2 of this specification. If their hardness is not more than 40 Rockwell C-scale and if they have been heated to 375° F ($\pm 10^\circ$) for 3 hours, hydrogen embrittlement shall be relieved in accordance with paragraph 3.3.14 or the coating must be removed by mechanical means. Following stripping, if parts are to be rephosphated, the parts shall be rinsed until the contiguous water film has a pH of 2 to 6, and if necessary to accomplish this condition an acidulated rinse per paragraph 3.3.3.2, A may be used.

NOTE: Attention is called to paragraphs 3.3.1 and 3.10 of Specification TT-C-490.

4. MANUFACTURING CONTROL PROVISIONS

4.1 General - Cleanliness shall be controlled by visual inspection. When parts prove to be unclean, they shall be reprocessed. All operations performed must be in accordance with the precautions given in paragraph 3.3.1.

4.2 Solution Control - All solutions shall be analyzed as often as required to maintain the concentrations stipulated in each cleaning method.

4.3 Finish - The finish of cleaned surfaces must not exceed in roughness the surface finish specifications of the original part when reworked parts are processed.

4.4 Sequence of Operations - The sequence of cleaning operations is governed by the job to be done, and no restrictions shall operate as long as the cleaning methods are not in conflict with the content of this specification.

4.5 Cleanliness - The cleanliness of finished parts shall be governed by the appropriate specification related to the service of the part.

5. PREPARATION FOR DELIVERY

Not applicable to this specification.

6. NOTES

6.1 Intended Use - This manufacturing specification, developed by the PE Laboratory of the George C. Marshall Space Flight Center, is intended for use in the cleaning of carbon steel alloys used in space launch vehicles.

6.2 Personal Safety - All necessary precautions regarding toxicity and industrial health hazards shall be taken by personnel engaged in cleaning ferrous parts in accordance with MSFC instructions and regulations. These precautions shall include ventilation over tanks and around mechanical cleaning methods and safety in the handling of chemicals or power tools.

6.3 Chemical Storage - Chemicals shall be stored in cabinets or storage rooms which can be maintained in a clean and orderly condition and which can be easily flushed with water to wash away or to neutralize spillage.

6.4 Handling Containers - Containers used in the transfer of chemicals shall be of materials not easily corroded or attacked. They shall be thoroughly washed and cleaned after usage.

NOTICE: When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING PROCESS DATA FOR
CLEANING OF MILD STEEL BOTTLE WITH SINGLE OPENING

1. SCOPE

This manufacturing process data covers the cleaning of mild steel bottles with a single opening.

2. EQUIPMENT AND MATERIALS

2.1: Equipment

- Holding Tool for steel bottle
- Tubing and connectors
- Valves
- Filters
- Spray lance

2.2 Materials

- CEE-BEE MX 15
- Freon
- Missile Grade Air
- Inert Gas

3. PROCEDURE

3.1 Cleaning - The mild steel bottle shall be placed in a vertical position with its opening facing downward.

3.1.1 Connect the bottle with the equipment provided by R-ME-P for spray operation in the Saturn I cleaning tower.

3.1.2 Spray with CEE-BEE MX-15 solution, 1.0 to 2.0 ounces per gallon of water, premixed in a tank, at 130° to 140° F for about 30 to 60 minutes.

3.1.3 Dry with missile grade air at 180 to 200° F for at least 1 hour.

3.1.4 Spray rinse with Freon to lowest particle count possible.

NOTE: No design requirements for particles. Best effort only required.

3.1.5 Dry with missile grade air (see Paragraph 3.1.4).

4. CONTROLS

4.1 Personnel shall insure that the following controls are exercised as applicable. Fill interior with inert gas as called out on work order and plug opening to retain the gas.

MANUFACTURING PROCESS DATA FOR
CLEANING OF MILD STEEL BOTTLE
WITH MULTIPLE OPENINGS

1. SCOPE

This manufacturing process data covers the cleaning of mild steel bottles with multiple openings.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Holding tool for steel bottle
- Tubing and connectors
- Valves
- Filters

2.2 Materials

- (a) Turco Alkaline Rust Remover, T-4181 or Turco 4008
- (b) Wayandotte Altrex
- (c) CEE-BEE-MX-15
- (d) Missile Grade air
- (e) Freon PCA
- (f) Inert gas

3. PROCEDURE

3.1 Preclean in Building 4760

3.1.1 Immerse bottle in Turco Alkaline Rust Remover T-4181 solution. 25 (±10) ounces per gallon of water at 200 to 212°F for 30 minutes or immerse bottle in Turco 4008 solution, full strength, at 270 to 275° F for 45 (±15) minutes.

- 3.1.2 Remove bottle from solution and allow it to drain.
- 3.1.3 Immerse bottle in Wayandotte Altrex solution, 8 ounces per gallon of water at 180 to 190° F, and fill and empty alternately at least three times.
- 3.1.4 Dry bottle with missile grade air at 180 to 200° F for about 1 hour.
- 3.1.5 Transfer the bottle to the Saturn I Cleaning Tower.

3.2 Clean in Saturn I Cleaning Tower

- 3.2.1 The bottle shall be placed in a position so that one opening will be facing downward in order that all the liquid will drain from it.
- 3.2.2 Convert the bottle with the equipment provided by R-ME-P to circulate the solutions back to the storage tank.
- 3.2.3 Recycle CEE-BEE-MX-15 solution, 1.0 to 2.0 ounces per gallon of water, premixed in a tank, at 140 to 160° F for from 30 to 60 minutes to a pH of about 6.6 to 7.6.
- 3.2.4 Dry with missile grade air at 180 to 200° F for about 1 hour.
- 3.2.5 Spray rinse with Freon to the lowest particle count.

NOTE: No design requirements for particles. Best effort only required.

- 3.2.6 Dry with missile grade air at 180 to 200° F.

4. CONTROLS

- 4.1 Personnel shall insure that the following controls are exercised as applicable:
 - 4.1.1 Fill interior with inert gas as called out on work order and plug the openings to retain the gas.
 - 4.1.2 Repaint exterior with semi-gloss white paint.

MANUFACTURING PROCESS DATA FOR
MILD STEEL CONTAINERS AND TUBULAR ASSEMBLIES
CHEMICAL CLEANING OF

1. SCOPE

1.1 Scope - This manufacturing procedure covers the necessary precautions and operations for chemical cleaning of containers and tubular assemblies composed of mild steel. The cleaning process is applicable to inside surfaces only. This procedure applies only to equipment assemblies composed of new materials. If assemblies being cleaned are excessively corroded or rusty, mechanical cleaning methods may be applied (abrasive blasting or wire brushing) prior to chemical cleaning as described in this report.

1.2 Applicability - This cleaning procedure will not apply to tubular assemblies subjected to excessive pressure unless properly heat treated.

2. EQUIPMENT AND MATERIAL

2.1 Equipment

2.1.1 Pump Unit (or cleaning console)

2.1.2 Electrical Source

2.1.3 Steam supply source

2.1.4 Hoses

2.1.5 Filters

2.1.6 Fittings and Connectors

2.1.7 High Pressure, High Flow Missile Grade Air Source

2.1.8 Dewpointer for Dewpoint Check

2.1.9 pH Meter (or pH testing paper)

2.1.10 Thermometers

- 2.1.11 Safety chains
- 2.1.12 Storage area
- 2.2 Materials
 - 2.2.1 Demineralized water
 - 2.2.2 Missile Grade Air
 - 2.2.3 Trichloroethylene
 - 2.2.4 Dow F-33 (or equivalent)
 - 2.2.5 Trisodium phosphate
 - 2.2.6 Hydrochloric Acid
 - 2.2.7 CEE-BEE MX-15 (rust inhibitor) (or equivalent)
 - 2.2.8 Solvent Precision Cleaning Agent, Trichloroethylene (Freon PCA) (or equivalent).

3. CLEANING

3.1 Chemical Processing

- 3.1.1 Attach or install the necessary flanges, adapters, and other cleaning equipment as necessary.
- 3.1.2 Solvent degrease with reclaimed or new trichloroethylene.
- 3.1.3 Dry by purging with missile grade air until no odor of trichloroethylene is detected at effluent points.
- 3.1.4 Detergent clean with an aqueous solution composed of 1 ounce of Dow F-33 (or equivalent) and 4 ounces of trisodium phosphate per gallon of water. The operating temperature of the solution shall be at least 120° F, and the period of contact shall be at least 1 hour.
- 3.1.5 Rinse with demineralized water until the effluent pH is between 6.0 and 8.0.

3.1.6 Acid clean with a 17 (± 5) percent by weight HCl solution. The solution will be used at ambient temperature, 85° ($\pm 10^\circ$) F. Period of contact will be as required to remove the rust.

3.1.7 The acid cleaning solution will be drained, and the rust preventative solution (CEE-BEE MX-15) will be pumped into the system until the volume of the system has been displaced at least twice. For spray application, the solution will be applied at sufficient pressure and flow rate to insure that all surfaces have been contacted.

NOTE: The rust preventative solution may be eliminated for equipment that is to be used in hydraulic service provided it can be filled with hydraulic oil before the surfaces rust. The hydraulic oil must meet the MSFC-PROC-166C Specifications.

In either case, flush until the effluent pH of the rust preventative solution is between 6.0 and 8.0, and the temperature of the effluent solution is not less than 140° F. Concentration of rust preventative solution will be 1 to 2 ounces of CEE-BEE M-15 (or approved equivalent) per gallon of demineralized water, and the temperature should be maintained at 150° F ($\pm 10^\circ$), (low solution operating temperature will not be caused for process interruption although the rust preventative solution is less effective at lower temperatures).

3.1.8 Purge with heated missile grade air until the system is dry.

NOTE: In accordance with MSFC-SPEC-164, the drying gas shall be prefiltered to zero (0) particles greater than 100 micron level (absolute), and oil content shall not exceed 3 parts per million (3 ppm) and the dewpoint shall be no higher than -65° F.

3.2 Testing

NOTE: The testing may be performed on the hydraulic oil which may be circulated through the tubing or the vessel to determine whether or not the conditions of MSFC-PROC-166C are satisfied.

3.2.1 A visual inspection of all openings will be made to insure that the component or system is free of all rust, corrosion, and other foreign contamination.

3.2.2 The component or system will be tested for particulate contamination by flushing thoroughly with solvent, precision cleaning agent as described

in MSFC-SPEC-237A Freon PCA, or the equivalent, and collecting a 500 ml portion of the test solution for testing in accordance with MSFC-SPEC-164 or MSFC-PROC-166C, as required.

3.2.3 Continue purge as in paragraph 3.2.1 until the effluent dewpoint is -65°F .

3.3 Packaging - Seal the component or system with flanges, fittings, or as specified in MSFC-SPEC-164, Cleanliness of Component for use in Oxygen, Fuel, and Pneumatic Systems and/or MSFC-PROC-166C, Hydraulic System Detail Parts, Components, Assemblies and Hydraulic Fluids for Space Vehicles, Cleaning Testing and Handling. Seal with cloth-backed tape that meets Fed Spec. PPP-T-60b Class I.

SECTION IX. MISCELLANEOUS MATERIALS

9. MISCELLANEOUS MATERIALS

The following sections present the chemical processes for materials that have been processed other than those previously classified in this manual.

9.1 TEFLON

Manufacturing Process - For the Cleaning of Teflon Lined Flux Hose - M-ME-MPROC 106.0

Cleaning and Application for Primer and Teflon Coating the OWS Meteoroid Shield Panels - MPD 49008

9.2 COPPER

Solder Coating Copper Clad Printed Circuit Boards Dip and Spin Method - MPD 28503

Cleaning of Copper for Photo Sensitive Resist Application - MPD 40009A

Cleaning of Printed Circuits (Copper Clad) Prior to Electroplating - MPD 40010A

Process Control and Operating Procedures for Copper Plating of Space Vehicle Parts - MPD 52001

9.3 SILVER

Manufacturing Process Data - For Cleaning of Tarnished Bifurcated Silver Terminal Boards - MPD 25011

Cleaning of ATM Silver Plated Components - MPD 26620

9.4 GOLD

Manufacture of Single and Double Sided Gold-Plated Printed Circuit Boards - MPD 28500

**Cleaning and Gold Plating Aluminum Alloys Specific for M512 Space
Experiments - MPD 52006A**

ADHESIVELY BONDED STRUCTURES

**Manufacturing Specification - For Fabricating of Adhesively Bonded
Honeycomb Structures Using Core and Face Sheets - M-ME-MPROC 005.5B-1**

**Structural Adhesive Bonding Metal to Metal Structures - M-ME-MPROC
005.5B-2**

**Manufacturing Specification - Cleaning for Adhesive Bonding -
M-ME-MPROC 005.5B-3**

**Manufacturing Process for Preparing Substrates for Adhesive Bonding-
M-ME-MPROC 005.5B-4**

MANUFACTURING PROCESS
FOR THE CLEANING OF TEFLON LINED FLUX HOSE

1. SCOPE

1.1 Scope

 This manufacturing process covers the approved Process Engineering Laboratory procedures for the cleaning of teflon lined flex hose.

1.2 Applicability

 The requirements set forth in this process are applicable to teflon lined flex hose to meet cleaning requirements of MSFC SPEC-164, MSFC-PROC-166, and 10419906.

2. APPLICABLE DOCUMENTS

2.1 Governmental

 The following documents, of the issue in effect on the date of use of this process, form a part of this process.

SPECIFICATIONS

Federal

O-S-642	Sodium, Phosphate, Tribasic, Technical Anhydrous, Dodecahydrate, and Mono-hydrate
O-T-634	Trichloroethylene
BB-N-411	Nitrogen
O-E-760	Ethyl Alcohol (Ethanol); Denatured Alcohol; and Proprietary Solvent

George C. Marshall Space Flight Center

MS 101.1	Drying Components in a Vacuum
MSFC-SPEC-237	Solvent "Freon" Precision Cleaning Agent, Specification for
MSFC-PROC-166	Cleaning, Testing and Handling of Onboard Hydraulic System Components and MIL-H-5606 Hydraulic Fluid, Procedure for
M-ME-MPROC 105.0	Manufacturing Process for Cleaning and Handling of Onboard Hydraulic System Components
MSFC-SPEC-164	Cleanliness of Components for Use in Oxygen, Fuel and Pneumatic Systems, Specification for
10419906	Cleaning of Components for Use in Gyro Air Supply Systems, Process for

3. REQUIREMENTS

3.1 Materials

3.1.1 Cleaning Solvents

- Demineralized Water - Demineralized water, when required for rinsing operations, shall be prefiltered to remove all particles greater than 175 microns in size in any dimension. The minimum specific resistance shall be 50 000 ohms and the pH shall be between 6 and 8.

- Trichloroethylene - Trichloroethylene, Types I and II, conforming to Specification O-T-634 shall be used.

- Drying and preservation gases - Gases used in drying and preservation processes shall be checked for moisture content and particulate contamination. The dewpoint shall be -65°F maximum. Particle size shall not exceed 20 microns in any dimension.

- Hydrion paper or pH indicating paper

3.2 Equipment

- pH meter
- Vapor degreaser
- Tube-cleaning console (Trisodium phosphate circulation system)
- Type I trichloroethylene flush system
- Source of heated drying media
- Test equipment for particle contamination and NVR

3.3 Procedure for Cleaning Teflon Lined Flex Hose

3.3.1 Process I - Fuel and Onboard Pneumatic Lines

3.3.1.1 Brush the "B" nuts with Type II trichloroethylene using a nylon bristle brush. Flush or spray the "B" nuts and the inside of the hose assembly with Type II trichloroethylene, utilizing the pump on the vapor degreaser.

3.3.1.2 Dry with oil-free, pre-filtered air or nitrogen at ambient temperature for a minimum of 5 minutes or until completely dry.

3.3.1.3 Circulate a 10 ± 2 percent solution of trisodium phosphate at $180^\circ \pm 10^\circ \text{F}$ for 15 to 20 minutes.

3.3.1.4 Rinse with deionized water until pH of effluent water is in the range of 6 to 8. Sample the effluent rinse for particle count.

3.3.1.5 Dry in accordance with paragraph 3.3.1.2.

3.3.2 Process II - Ground Pneumatic and LOX lines

3.3.2.1 Brush the "B" nuts with Type II trichloroethylene using a nylon bristle brush. Flush or spray the "B" nuts and the inside of the hose assembly with Type II trichloroethylene, utilizing the pump on the vapor degreaser.

3.3.2.2 Dry in accordance with paragraph 3.3.1.2.

3.3.2.3 Circulate a 10 ± 2 percent solution of trisodium phosphate at $180^\circ \pm 10^\circ \text{F}$ for 15 to 20 minutes.

3.3.2.4 Rinse with deionized water until pH of effluent water is between 6 and 8.

3.3.2.5 Dry in accordance with paragraph 3.3.1.2.

3.3.2.6 Flush with trichloroethylene, Type I, sample the used trichloroethylene for particle count, per the Millipore method, and also sample the trichloroethylene before and after passing through the flex hose for nonvolatile residue per paragraph.

3.3.2.7 Nonvolatile Residue (Except non-metallic materials)

The used and unused test solvent, trichloroethylene or equivalent, collected in the clean container shall be examined for nonvolatile residue as follows:

(a) Evaporate the solvent to 10 to 20 ml. volume in a steam bath.

(b) Transfer the solvent to a constant weight (within 0.3 mg.) tared 30 ml. weighing to the nearest 0.1 mg.

(c) Continue the evaporation by placing the weighing bottle inside a constant temperature oven which has normalized at 221° to 230°F . Allow weighing bottle to remain inside the oven for a maximum of 1.5 hours or until the solvent is evaporated to dryness.

(d) Remove weighing bottle from the oven and place in a desiccator for cooling.

(e) After cooling, remove the weighing bottle from the desiccator and weigh the bottle to the nearest 0.1 mg.

(f) Return the weighing bottle to the normalized, constant temperature oven at 221° to 230°F for 0.5 hour.

(g) Repeat step (d) and step (e). If the difference in the weights taken in step (e) and step (g) is greater than 0.3 mg., repeat step (f), (d), and (e) until the difference in the weighings is 0.3 mg. or less.

(h) Compare the test results obtained from the used solvent to the results obtained from the unused solvent for conformance to the cleanliness level specified herein.

3.3.2.8 Dry in accordance with paragraph 3.3.1.2.

3.3.3 Process III - Sloss Measuring and Gas Bearing System Lines

3.3.3.1 Brush the "B" nuts with Type II trichloroethylene using a nylon bristle brush. Flush or spray the "B" nuts and the inside of the hose assembly with Type II trichloroethylene, utilizing the pump on the vapor degreaser.

3.3.3.2 Dry in accordance with paragraph 3.3.1.2.

3.3.3.3 Circulate a 10 ± 2 percent solution of trisodium phosphate at $180^\circ \pm 10^\circ$ F for 15 to 20 minutes.

3.3.3.4 Rinse with deionized water until pH of effluent water is between 6 and 8.

3.3.3.5 Dry in accordance with paragraph 3.3.1.2.

3.3.3.6 Flush with trichloroethylene, Type I, in accordance with paragraph 3.3.2.6, less particle count.

3.3.3.7 Dry in accordance with paragraph 3.3.1.2.

3.3.3.8 Nonvolatile Residue - Examine for nonvolatile residue in accordance with paragraph 3.3.2.7.

3.3.3.9 Flush with pre-filtered alcohol and sample alcohol for particle count per Millipore method.

3.3.3.10 Dry - Allow to air dry in checkout room.

3.3.4 Process IV - Hydraulic Lines

3.3.4.1 Brush the "B" nuts with Type II trichloroethylene using a nylon bristle brush. Flush or spray the "B" nuts and the inside of the hose assembly with Type II trichloroethylene, utilizing the pump on the vapor degreaser.

3.3.4.2 Dry in accordance with paragraph 3.3.1.2.

3.3.4.3 Circulate a 10 ± 2 percent solution of trisodium phosphate at $180^\circ \pm 10^\circ\text{F}$ for 15 to 20 minutes.

3.3.4.4 Rinse with deionized water until pH of effluent water is between 6 and 8.

3.3.4.5 Flush with alcohol to remove water, then sample alcohol for particle count per Millipore method.

3.3.4.6 Dry in accordance with paragraph 3.3.3.10.

4. ACCEPTABILITY PROVISIONS

4.1 Acceptability

The cleanliness levels of teflon lined flex hoses shall meet the particle and nonvolatile residue requirements of MSFC-SPEC-164, MSFC-PROC-166, and Dwg. No. 10419906 as applicable.

NOTE

(1) This manufacturing process has been coordinated with the Process Engineering Laboratory and is intended for use in cleaning, processing, and testing of Teflon lined flex hose. The final verification of this procedure will be established during the production of the vehicle.

(2) Any deviation from this basic procedure will require written concurrence from the Process Engineering Laboratory. An emergency concurrence may be made by telephone, but it must be confirmed in writing.

PREPARING ACTIVITY

Process Engineering Laboratory
George C. Marshall Space Flight Center

CLEANING AND APPLICATION FOR PRIMER AND
TEFLON COATING THE OWS METEOROID SHIELD
PANELS

1. SCOPE

This specification covers the procedure for conversion coating, priming and teflon coating the meteoroid panels.

2. DOCUMENTS

2.1 Federal

2.1.1 Federal Test Method Std. 141, Method 6301, Paragraph 2.1.

3. REQUIREMENTS

3.1 Facilities

3.1.1 Building 4760, Surface Treat and Paint Shop.

3.1.1.1 Missile grade air.

3.1.2 Building 4707, Large Walk-In Oven.

3.1.3 Adequate ventilation.

3.1.4 Paint Shop, Building 4760, shall be cleaned free of visible dust or dirt before applying coatings.

3.1.5 Unless otherwist specified, apply coatings at room temperature above 50°F and relative humidity below 80 percent.

3.2 Chemicals

3.2.1 Acetone.

3.2.2 MEK.

3.2.3 Freon.

- 3.2.4 99 percent olefin free grade of hexadecane.
- 3.2.5 Ethylene glycol monoethyl ether (cellosolve).
- 3.2.6 Existing chemical solutions in Building 4760.
- 3.2.7 Super koropon-R-515-700 and R-910-704 epoxy primer (green color).
- 3.2.8 Teflon-S 954-101 (light green color).
- 3.2.9 Teflon-S thinner T-8748.
- 3.2.10 Teflon clean-up solvents T-8748 and/or T-8754.

3.3 Spray Equipment

- 3.3.1 Binks spray gun, model 18 with number 63 needle, number 63 fluid nozzle and number 66SD air cap or equivalent equipment.
- 3.3.2 Binks spray gun, model 18 with number 63 needle, 63B fluid nozzle, 63 PB air cap or equivalent equipment.
- 3.3.3 Additional number 63A needle, number 63 PB air cap and number 63C fluid nozzle for primer application.
- 3.3.4 Zahn viscometers.
- 3.3.5 Dry film thickness testers.
- 3.3.6 Paint mixers.

3.4 Materials

- 3.4.1 Polyethylene, 6 mils.
- 3.4.2 Nylon-6, 2 to 6 mils.
- 3.4.3 600 grit aluminum oxide sand paper.
- 3.4.4 Rubber gloves.

- 3.4.5 Respirators.
- 3.4.6 Brown craft paper.
- 3.4.7 Sponges, cheese cloth or diapers for wipe cleaning.
- 3.4.8 Paint strainer, filter, or 150 mesh wire screen.
- 3.4.9 Panel, 8 feet x 10 inches x 1/8 inch aluminum test panels.
- 3.5 Controls
- 3.5.1 Koropon primer R-515-700 and R-910-711.
- 3.5.1.1 Adhesion Test - Reference Paragraph 3.7.1.1.
- 3.5.1.2 Thickness - The dry film thickness shall range from 0.7 to 1.0 mil.
- 3.5.1.3 Pot Life of Mixed Primer - The pot life of the catalyzed primer shall last up to 8 hours at 77° F; however, pot life is shorter at higher temperatures.
- 3.5.1.4 Solids Content - The solids content of the catalyzed primer shall be 38 ± 2 percent. (NOTE: If Primer is purchased under a Douglas Drawing specification, the minimum solids content is 30 percent.)
- 3.5.1.5 Mixing Ratio for primer shall be on a one to one ratio. NOTE: It is important to have the correct ratios by volume.
- 3.5.1.6 Viscosity shall be tested with Zahn No. 2.
- 3.5.2 Teflon-S-954-101.
- 3.5.2.1 Bead test for coalesced surfaces - reference paragraphs 3.7.2.2.C.
- 3.5.2.2 Thickness - The dry film thickness shall range from 1.0 to 1.5 mils unless otherwise specified.
- 3.5.2.3 Solids - The solid contents of the Teflon-S shall be 51.9 ± 2 percent.

3.5.2.4 Viscosity - The viscosity shall range from 43 to 53 with number two Zahn viscometer.

3.6 Conversion Coated Parts

Parts shall be air dried 18 to 24 hours before painting.

3.7 Tests

3.7.1 For koropon epoxy primer.

3.7.1.1 Adhesion Test (NOTE: Allow 72 hours minimum drying times at room temperature). Apply a strip of 1 inch wide masking tape to the test panel and press down by rolling with 4 1/2 pound number roller 8 times; four times alternating in each direction.

A. Testing - Grasp the tape by one end and remove it with an abrupt motion, maintaining an angle of approximately 90 degrees.

B. Examine the tested area for paint damage, such as removal of paint to the conversion coat or base material.

C. Failure - A failure is noted when more than 1 square inch of primer is removed.

D. Action - Strip and reprocess per paragraph 5.

3.7.1.2 Solids - Heat the calalyzed primer for two hours at 200° F, then determine the weight loss.

3.7.2 Teflon-S for cured film.

3.7.2.1 The Teflon-S coating shall have a coalesced surface.

3.7.2.2 Method - Bead tracking test.

A. The test panel shall be cleaned with an aqueous solution of 1 to 2 percent Triton X-100.

B. Rinse - The surface shall be rinsed for 5 to 6 times with D. I. water and placed in a horizontal position.

C. Testing for a coalesced surface - Two or three drops of hexadecane shall be dropped from a pipett from a height of 1/2 inch onto the coated surface. A bead should form. In addition, tilt the panel so the bead may run off.

D. Failure - A failure shall occur when a bead does not form or when a trail is left after tilting the panel.

3.7.2.3 Solids - Solids shall be run on a sample of Teflon S (liquid) for 30 minutes at 450° F.

3.8 Test Panels

Test panels shall be run parallel with spray parts.

4. PROCEDURES - CONVERSION COATING OF ALUMINUM

4.1 Hand Clean

Parts shall be hand cleaned with a cloth moistened with Acetone to remove dirt, grease or ink.

4.2 Degrease

The parts shall be vapor degreased in Trichloroethylene until vapors cease to condense on parts.

4.3 Alkaline Clean

The parts shall be immersed in Turco 4215 at 180° F ($\pm 10^\circ$) for 45 \pm 15 minutes.

4.4 Rinse

The parts shall be spray rinsed as parts are removed from Alkaline cleaning before rinsing in the hot water rinse tank for 15 to 10 minutes at 180° F ($\pm 10^\circ$).

4.5 Deoxidize

The parts shall be deoxidized in Turco 2897 at room temperature for 30 \pm 15 minutes.

4.6 Rinse

The parts shall spray rinse as parts are being removed from deoxidizer before rinsing in cold water rinse for 5 to 10 minutes.

4.7 Conversion Coat

The parts shall be conversion coated in an agitated solution of Iridite 14-2 from 10 seconds to 90 seconds at room temperature depending upon the alloy.

4.8 Rinse

The part shall be spray rinsed as parts are removed from Iridite 14-2 before rinsing in the cold water rinse at room temperature for 5 to 10 minutes.

4.9 Dry

The parts shall be air dried for a minimum of 18 to 24 hours before any painting occurs.

4.10 Package

Parts shall be wrapped with brown craft paper or polyethylene before storing or routing to other areas.

5. PROCEDURES PRIMING

5.1 Preclean

The surfaces shall be wiped with a cloth moistened with acetone or MEK. (NOTE: If 12 hours elapses before priming, repeat solvent wipe)

5.2 Primer

The panels and/or parts shall be primed with super koropon resistant primer. (NOTE: Filter primer before using).

5.2.1 Viscosity - The viscosity of the primer shall vary from 27 to 33 seconds.

5.2.2 Thickness (dry) - The primer thickness shall vary from 0.7 to 1.0 mils.

5.2.3 Cure of primed parts.

5.2.3.1 Room temperature cure, or paragraph 5.2.3.2.

A. Dust free - The primer shall be dust free within 5-10 minutes after application.

B. Tack free - The primer shall be tack free within 30-45 minutes after application.

C. Full Cure - The primer shall be fully cured for further coatings after 24 hours.

5.2.3.2 Elevated temperature cure - The primer shall be cured at 200°F for 1 to 1 1/2 hours, for further coating application.

5.2.4 Additional coating - An additional coating may be applied at 1 hour at 77° F as applied to paragraph 5.2.3.1.

6. PROCEDURE FOR TEFLON COATING

6.1 Surface preparation

The primed surface of panels shall be sanded with 600 grit aluminum oxide paper until the surface is smooth to the touch of the hand, or as otherwise specified.

6.2 Clean

The surface shall be wiped with cloths moistened with acetone or MEK until there is no discoloration and allow to dry for minimum of 1 hour.

6.3 Mixing Teflon-S

The Teflon-S shall be mixed at the specified viscosity in a light mixer or blend at low to moderate speeds.

6.3.1 Filter - The liquid shall be filtered through a paint strainer or 150 mesh wire screen.

6.4 Application

6.4.1 Spraying - The meteoroid shield panels shall be sprayed with 954-101 Teflon-S as specified by drawings.

NOTE: Avoid contaminating other parts with over spray. Wear respirators.

6.4.2 Viscosity - The viscosity of teflon material shall range from 43 to 53 with Zahn #2.

6.4.3 Thickness - Film thickness shall range from 1.0 to 1.5 or otherwise specified.

NOTE: One spray pass produces approximately 1.2 mils dried film.

6.4.4 Cure time

NOTE: Panels shall be cured in walkin oven in building 4707.

6.4.4.1 For multiple coatings - The sprayed, coated panels shall be allowed to air dry for 3 to 4 hours* * before curing the initial coat at 300° F for 15 minutes. For additional coatings the same cure shall be repeated until final coating as above, which will be cured at 300° ± 10° F for 8 to 9 hours but total time above 200° F shall not exceed 9 hours.

NOTE: Teflon-S must be cured before 4 hours elapse at room temperature.

6.4.4.2 Cure times for Teflon-S.

<u>TIME</u>	<u>TEMPERATURE</u>
<u>20 minutes</u>	<u>450° F</u>
<u>13 minutes</u>	<u>475° F</u>
<u>9 minutes</u>	<u>500° F</u>
<u>5 minutes</u>	<u>525° F</u>

6.4.4.3 Cure time for meteroid shield panels shall be 350° ± 10° F for 8 to 9 hours but total time above 200° F shall not exceed 9 hours.

6.4.5 Thickness - After cooling spot check thickness on meteroid shield panel as well as test panels.

6.4.6 Package - The meteoroid shield and fixture shall be wrapped with polyethylene before transferring.

7. QUALITY ASSURANCE

7.1 Verification

The following paragraphs must be verified.

7.1.1	Reference paragraphs	3.5.1.1
7.1.2		3.5.1.2
7.1.3		3.5.1.3
7.1.4		3.5.1.4
7.1.5		3.5.1.5
7.1.6		3.5.1.6
7.1.7		3.5.2.1
7.1.8		3.5.2.2
7.1.9		3.5.2.3
7.1.10		3.5.2.4
7.1.11		3.8
7.1.12		5.2.2
7.1.13		6.4.4.3
		6.4.5

8. SAFETY PRECAUTIONS

8.1 Precautions

8.1.1 At the process temperatures, the vapors liberated from Teflon-S may be harmful and should not be breathed.

8.1.2 Solvents in both the primer and Teflon-S are combustible. Keep away from heat and open flame.

8.1.3 During mixing, spraying and curing operations the vapors should not be breathed.

NOTE: Respirators should be used during these operations.

8.1.4 Avoid prolonged contact with skin. Wash thoroughly after handling the materials.

8.1.5 Keep all containers closed at all times.

SOLDER COATING
COPPER CLAD PRINTED CIRCUIT BOARDS
DIP AND SPIN METHOD

1. SCOPE

This manufacturing procedure specifies the equipment, materials, pre-cleaning and post cleaning technique, and the operation of the dip and spin solder coating machine. This procedure is to be used when applying a thin, tightly adhering coating of fused tin-lead solder to a bare copper or solder plated printed circuit board by the dip and spin method.

2. REQUIREMENTS

Printed circuit boards processed in accordance with this procedure may have conductors on one or both sides, be of the multilayer type, and contain holes which are unsupported or plated through. Printed circuit boards to be processed by this procedure shall be manufactured in accordance with MSFC-STD-154, and shall have bare copper or tin-lead solder plated conductors.

3. EQUIPMENT AND MATERIALS

3.1 Precleaning equipment tanks

Tanks used for precleaning processes shall be of a size compatible with production requirements, and shall be equipped with an adequate ventilation system for removal of vapor fumes and odors.

3.1.1 Cleaning Tank

A tank constructed of No. 316 stainless steel containing a sump drain, and dump valve of similar material. The tank shall be equipped with a stainless steel immersion type heater capable of raising a solution filled tank to a maximum of 200° F during a 2 hour period, and maintaining a given temperature setting within $\pm 10^\circ$ F. The fixture from which materials are suspended shall be electrically insulated from the body of the tank.

3.1.2 Spray Rinse Tank

A tank constructed of No. 300 series stainless steel containing a drain

of similar material sufficient in size to quickly empty its contents. The tank shall be equipped with a spray system capable of simultaneously rinsing all sides of an immersed object.

3.1.3 Etch Tank

A tank constructed of polyethylene, polypropylene, or No. 316 stainless steel containing a drain of similar material.

3.1.4 Acid Pickle Tank

A tank constructed of polyethylene, polypropylene, or mild steel having an internal lining of PVC material and exterior corrosion protection. The fixture from which materials are suspended shall be electrically insulated from the body of the tank.

3.1.5 Tin Immersion Tank

A tank constructed of polypropylene, or mild steel having an internal lining of high temperature plastic and exterior corrosion protection. The tank shall be equipped with a low current density, quartz electric immersion heater capable of raising a solution filled tank to a maximum of 200° F during a 2 hour period, and maintaining a given temperature setting within $\pm 10^{\circ}$ F.

3.1.6 Deionized Water Hot-Rinse Tank

A tank constructed of No. 316 stainless steel or polypropylene, and equipped with an immersion heater similar to that of the tin immersion tank as specified in paragraph 3.1.5, and a dam or standpipe type overflow.

3.2 Post Cleaning Equipment Tanks

3.2.1 Hot Water Cleaning Tank

A tank constructed of stainless steel and equipped with an immersion heater similar to that specified in paragraph 3.1.1.

3.2.2 Ultrasonic Cleaning Tank

A tank constructed of stainless steel which is energized by an ultrasonic frequency generator.

3.3 Solder Coating Equipment

A solder coating machine employing the dip and spin method of operation shall be used.

3.4 Materials

3.4.1 Cleaner

Neutraclean No. 7, manufactured by the Shipley Company, Inc. 2300 Washington Street, Newton, Mass., or equivalent.

3.4.2 Deionized Water

Maximum conductivity: 50 micromhos; temperature: 70 to 90 degrees F; pressure: 60 to 100 psi.

3.4.3 Etchant

Ammonium persulfate, crystals, anhydrous.

3.4.4 Acid Pickle

Hydrochloric acid, 36.5 percent - 38 percent Reagent, ACS.

3.4.5 Tin Immersion

Shipley LT-26, manufactured by the Shipley Company, 2300 Washington Street, Newton, Mass., or equivalent.

3.4.6 Air

Missile grade, 40 to 60 psi pressure, for drying purposes.

3.4.7 Solder

63 percent tin - 37 percent lead, or 60 percent tin - 40 percent lead, per Federal Specification QQ-S-571

3.4.8 Solder Blanket Liquid

Aqua-Sol, manufactured by MK Associates, P. O. Box 746, Huntsville, Ala., or equivalent.

3.4.9 Flux

Soldering, liquid (rosin base) per MIL-F-14256, Kester 1544, or equivalent.

3.4.10 Degreaser

Freon, type TF, or equivalent.

4. PROCEDURE

4.1 Preparing Cleaning Chemical Solutions

4.1.1 Cleaning Tank Solution

Mix 12 ounces of Neutraclean No. 7 per 1 gallon of water.

4.1.2 Etch Tank Solution

Mix 2 pounds of Ammonium Persulfate per 1 gallon of water.

4.1.3 Acid Tank Solution

Ratio: 20 to 30 percent of volume Hydrochloric acid.

4.1.4 Tin-immersion Tank Solution

Ratio: 6.25 pounds of Shipley LT-26, 4 gallons of water, 1 quart of Reagent Hydrochloric acid.

4.2 Precleaning Printed Circuit Boards

4.2.1 Procedure for Bare Copper Conductors

4.2.1.1 Clean

(a) Immerse board into cleaning tank containing a solution of Neutraclean No. 7 that has been raised to operating temperature. Immersion time shall vary with solution temperature as follows:

130 degrees	10-12 minutes
150 degrees	5-8 minutes
180 degrees	3-5 minutes
200 degrees	2-3 minutes

- (b) Rinse board in spray rinse tank for 30 to 45 seconds.

4.2.1.2 Etch

(a) Immerse board into etch tank containing a solution of Ammonium Persulfate for approximately 3 seconds.

- (b) Remove board from solution and hold in the air.

(c) Repeat immersion and aeration for approximately 10 times, or until a uniform pink color appears on the copper conductor surfaces.

- (d) Rinse board in spray rinse tank for 30 to 45 seconds.

4.2.1.3 Acid Dip

(a) Immerse board into acid pickle tank containing a solution of Hydrochloric acid for 30 to 45 seconds continuously.

- (b) Rinse board in spray rinse tank for 30 to 45 seconds.

4.2.1.4 Condition Dip

(a) Immerse board into cleaning tank as specified in paragraph 4.2.1.1(a).

- (b) Rinse board in spray rinse tank for 30 to 45 seconds.

4.2.1.5 Tin Immersion Dip

(a) Immerse board into tin immersion tank containing a solution of Shipley LT-26 that has been raised to operating temperature. Immersion time shall be as follows:

150 degrees 5 minutes

180 degrees 3 minutes

- (b) Rinse board in spray rinse tank for 30 to 45 seconds.

(c) Immerse board into deionized water hot-rinse solution tank that has been raised to an operation temperature of 180°F (±10°) for 1 to 3 minutes.

CAUTION: Do not touch the tinned conductor surfaces of the board.

4.2.1.6 Flux

(a) Immediately after the tin immersion dip and drying (4.2.1.5d) has been completed, immerse board into a flux mixture, using caution that the board holding fixture does not come in contact with the tinned conductor surfaces.

(b) Place the board in a slot type holding rack. No part of the rack shall touch the tinned conductor surfaces.

(c) Allow the flux to air dry at room temperature, or force dry in a gravity convection oven at 150 °F (± 10°).

4.2.2 Procedure for Solder Plated Conductors

4.2.2.1 Clean

Proceed as directed in paragraph 4.2.1.1.

4.2.2.2 Acid Dip

Proceed as directed in paragraph 4.2.1.3.

4.2.2.3 Condition Dip

Proceed as directed in paragraph 4.2.1.4.

4.2.2.4 Tin Immersion Dip

Proceed as directed in paragraph 4.2.1.5.

4.2.2.5 Flux

Proceed as directed in paragraph 4.2.1.6.

4.2.3 Storage of Precleaned Boards

Precleaned circuit boards may be stored prior to the solder dip and spin process provided an adequate means of protection is used. The dried flux coating on the board surface shall not be removed or damaged. The storage area shall be kept clean and dry.

4.3 Solder Dipping and Spinning

4.3.1 Machine Preparation

The solder coating machine shall be prepared for operation as follows:

- (a) Turn on the solder pot power switch and allow the solder to become molten.
- (b) Introduce solid bars of solder into the pot until the molten solder level is approximately 1/2 inch below the top surface. Stir the liquid solder so that the metals are thoroughly diffused.
- (c) Dross the liquid surface and add 1/4 inch of solder blanket liquid.
- (d) Firmly seat the splash guard in the pot with the raised apron toward the solder pot access door.
- (e) Turn on air and adjust the pressure to 70 to 80 psi.
- (f) Turn on the machine motor power switch located at the rear of the machine. The loading door should open, and the drive motor should start.
- (g) Adjust the dip timer located inside the real control panel to a setting which allows maximum wetting action without overheating the board material. Average settings are 3 to 5 seconds at a solder pot temperature of 490° F ($\pm 10^\circ$).
- (h) Adjust the dip timer located adjacent to the dip timer. An average setting of 3 to 5 seconds will provide maximum acceleration. Longer settings will allow the fixture and board to cool to a lower degree.

4.3.2 Machine loading

The fixture shall be adjusted and loaded as follows:

- (a) Remove safety and depress top and bottom latch buttons allowing the gate to open.
- (b) Remove the 8 wing nuts and screws from side supports, and the stop assemblies from the side supports.

(c) With board in place and gate closed, move side supports into contact with both sides of board.

(d) Install screws and wing nuts loosely into left side support. Move board and side support laterally to locate nearest slotted holes for the right side support. Install right side support screws and wing nuts.

(e) Press the board firmly against the closed gate and tighten the side supports into place in contact with the board edge, allowing approximately 0.03 inch for thermal expansion.

(f) Install the stop assemblies on the side supports allowing approximately 0.03 inch total end clearance between the board and the stop.

(g) The fixture may now be loaded by opening the gate and inserting a board between the side supports.

CAUTION: To prevent serious damage to the fixture during the cycle operation, insure that the latching pins are fully extended through the latching holes located on gate, and the pin safety is in the closed position.

4.3.3 Machine Operation

The solder coating machine shall be operated as follows:

(a) Close the loading and solder pot access doors firmly.

(b) Depress the cycle button until the green light stays on. The loading door will lock automatically as the cycle begins. When the cycle is completed the green cycle light will go off and the door latch will open.

(c) Open the loading door and remove the board from the fixture.

NOTE: The interrupt pushbutton will stop the cycle at any time. The reset button located at the rear of the machine will clear the interrupt condition and return the fixture to the loading position.

4.4 Post Cleaning Printed Circuit Boards

Printed circuit boards that have been solder coated by the dip and spin process shall be cleaned as follows:

(a) Immerse board into the hot water cleaning solution tank that has been raised to an operating temperature of 180° F ($\pm 10^\circ$).

(b) Agitate board in the solution until the majority of the oil cover material is removed.

(c) Remove board and blow dry with air.

(d) Immerse board into ultrasonic cleaning tank containing a solution of Freon type TF for approximately 3 to 5 minutes.

CAUTION: Do not touch the board conductor surfaces after the ultrasonic cleaning process has been performed.

(e) Remove board and blow dry with air.

(f) Place board in a protective package.

5. QUALITY CONTROL

5.1 Printed Circuit Board Inspection

An inspection of the solder coated printed circuit board shall be performed after the post cleaning process. The following coating defect shall not be present:

(a) Incomplete coating.

(b) Grey or mottled coating.

(c) Rough coating.

(d) Plugged or uncoated holes.

5.2 Control of Chemical Solutions

(a) 4.1.1 Neutraclean No. 7. Dump when solution will not remove oxides within 1 minute tank temperatures of 150 ° F or above.

(b) 4.1.2 Ammonium Persulfate: Make up new tank daily.

(c) 4.1.3 Hydrochloric Acid: Analyze weekly and hold solution at 20 to 30 percent concentration. Dump solution when it becomes milky or yellowish in color.

(d) 4.1.4 Shipley LT-26: Follow manufacturer's instructions.

CLEANING OF COPPER FOR
PHOTO SENSITIVE RESIST APPLICATION

1. SCOPE

1.1 Scope

This procedure details the equipment, materials, production techniques and quality control requirements to be utilized when cleaning copper clad printed circuit material prior to the application of a photo sensitive resist. Where mechanical cleaning is performed such cleaning shall be prior to the cleaning techniques specified herein.

1.2 Application

Copper clad printed circuit material(s) cleaned in accordance with this procedure shall be coated with photo sensitive resist within 1 hour after the specified cleaning procedure taking care not to expose or otherwise contaminate the cleaned surface with dust, moisture or other detrimental contaminants.

2. EQUIPMENT AND MATERIALS

2.1 Cleaning Equipment Tanks

Tanks used for cleaning procedures shall be of a size compatible with production requirements and shall be equipped with an adequate ventilation system for removal of vapor fumes and odors.

2.1.1 Cleaning Tank

A tank constructed of #316 stainless steel containing a sump drain and dump valve of similar material. The tank shall be equipped with a stainless steel immersion type heater capable of raising a solution filled tank to a maximum of 200 °F during a 2 hour period, and maintaining a given temperature setting within $\pm 10^\circ\text{F}$. The fixture from which materials being cleaned are suspended shall be electrically insulated from the body of the tank.

2.1.2 Spray Rinse Tank

A tank constructed of #300 series stainless steel containing a drain of similar material sufficient in size to quickly empty the tank contents. The tank shall be equipped with a spray system capable of simultaneously rinsing all sides of an immersed object.

2.1.3 Etching Tank

A tank constructed of polyethylene, polypropylene or #316 stainless steel containing a drain and valve of similar material.

2.1.4 Acid Dip Tank

A tank constructed of polyethylene polypropylene or of mild steel having an internal lining of PVC material and the exterior suitably protected from corrosion. The fixture from which the printed circuit material being cleaned is suspended shall be electrically insulated from the body of the tank.

2.1.5 Deionized Water Rinse Tank

A dip rinse tank of polypropylene, polyethylene or of No. 316 stainless steel containing a drain and valve of similar material and either a dam or standpipe type overflow.

2.1.6 Anti-Tarnish Soak Tank

A dip tank constructed of polyethylene, polypropylene or of No. 316 stainless steel containing a drain and valve of similar material. Tank shall be of sufficient size to allow for an immersion time of approximately 15 minutes.

2.2 Materials

2.2.1 Cleaner

Neutralclean No. 7, manufactured by Shipley Company, 2300 Washington St., Newton, Mass.

2.2.2 Deionized Water

Maximum conductivity 50 micro-ohms, temperature 70 to 90°F, pressure 60 to 100 psi.

2.2.3 Etchant

Ammonium Persulfate, crystals, anhydrous

2.2.4 Acid Dip

Hydrochloric Acid, 36.5 to 38 percent, Reagent A.C.S.

2.2.5 Anti-Tarnish Dip

Entek CU-55, manufactured by Enthone, Inc., New Haven, Conn.

2.2.6 Missile Grade Air

40 to 60 psi pressure for drying purposes.

3. PROCEDURE FOR TANK MAKE UP

3.1 Clean (organic removal)

Mix 12 ounces of Neutraclean No. 7 per 1 gallon of deionized water.

3.2 Etch

Mix 2 pounds of Ammonium Persulfate per 1 gallon of deionized water. The use of warm water after the initial addition of cold water to the tank is recommended in order to rapidly obtain the operating temperature of 70 to 90°F desired since Ammonium Persulfate upon dissolving tends to produce considerable solution cooling.

3.3 Acid Dip

Add 20 to 30 percent by volume of Hydrochloric Acid to Deionized water to operating volume.

3.4 Anti-Tarnish Soak

37 Milliliters of Entek CU-55 per 1 gallon of water (D.I.)

4. PROCEDURE FOR CLEANING FOR RESIST APPLICATION

4.1 Clean

(a) Immerse board into cleaning tank containing a solution of Neutraclean No. 7 that has been raised to operating temperature. Immersion time shall vary with solution temperature as follows:

130 degrees F	10-12 minutes
150 degrees F	5-8 minutes
180 degrees F	3-5 minutes
200 degrees F	2-3 minutes

(b) Rinse board in spray rinse tank for 30 to 45 seconds.

4.2 Etch

(a) Immerse board into etch tank containing a solution of Ammonium Persulfate for approximately 3 seconds.

(b) Remove board from solution and hold in air for approximately 3 seconds.

(c) Repeat steps (a) and (b) ten times, or until a uniform pink color appears on the copper surface(s). Immersion time should not exceed 40 seconds total in the solution in order to prevent removal of excessive copper.

(d) Rinse board in spray rinse for 30 to 45 seconds.

4.3 Acid Dip

(a) Immerse board completely in acid dip tank containing hydrochloric acid for a continuous period of 30 to 45 seconds.

(b) Dip and move about in the dip rinse tank for a period of 15 to 20 seconds.

4.4 Anti-Tarnish Dip

Completely immerse in the Entek CU-55 solution for a period of 12 to 15 minutes. Do not rinse afterwards.

4.5 Dry

Blow both sides of the copper clad laminate dry with a blast of missile grade air. Make sure to handle the copper clad board by the edges only during the drying operation and all subsequent operations prior to the coating of the panel with photo resist. The appearance of water breaks on the surface when the panel is removed from the anti-tarnish dip is probable but not always present when all cleaning procedures have been correctly performed.

5. QUALITY CONTROL

5.1 Control of Chemical Cleaning Solutions

5.1.1 Cleaner

Neutraclean No. 7 - Dump when solution will not remove oxides within 1 minute when tank temperature is 150°F or above. Dump tank weekly on a regular schedule basis.

5.1.2 Etch

Ammonium Persulfate - make up new bath daily. Make sure that the bath temperature is at least 70°F before using. The use of hot water to bring up the temperature is permissible when added after cold water starts the dissolving action of the crystals.

5.1.3 Acid Dip

Hydrochloric Acid - Laboratory analysis at least weekly holding solution concentration between 20 percent and 30 percent by volume. Dump solution when solution becomes cloudy, milky or tinged with yellow.

5.1.4 Anti-Tarnish Dip

Entek CU-55. Dump solution when adhesion test(s) as described below are unsatisfactory and at least on a weekly basis. When work load is light laboratory analysis on a regular basis should be substituted for the regular dumping schedule.

5.2 Control of Cleaning by Resist Adhesion Testing (Optional Method)

After application of photosensitive resist, ultraviolet vacuum frame printing, developing, dyeing and post drying test the resist adhesion with a strip of Minnesota Mining and Manufacturing Company's Scotch No. 470

electroplaters tape by adhering a strip of the tape to the resist pattern, rubbing the tape briskly to warm the tape and give good tape adhesion and then jerk the tape from the surface. The appearance of any dyed portion of the photo resist on the tape indicates that cleaning was not sufficient. This test is particularly important when duplex (i. e., gold plated contacts and solder plated pattern) electroplating is to be performed.

CLEANING OF PRINTED CIRCUITS (COPPER CLAD) PRIOR TO ELECTROPLATING

1. SCOPE

1.1 Scope - This procedure details the equipment, materials, production techniques and quality control requirements to be utilized when cleaning of copper clad printed circuit boards (photo sensitive resist applied to the copper surfaces) prior to electroplating of the circuit.

1.2 Application - Copper clad printed circuit material coated with photo sensitive resist cleaned in accordance with this procedure shall be transferred to the appropriate electroplating tank within 30 seconds following initiation of rinsing and electroplating initiated upon starting into the tank.

2. EQUIPMENT AND MATERIALS

2.1 Cleaning Equipment Tanks - Tanks used for cleaning procedures shall be of a size compatible with production requirements and shall be equipped with an adequate ventilation system for removal of vapor fumes and odors.

2.1.1 Cleaning Tank (organic removal) - A tank constructed of No. 316 stainless steel containing a sump drain and dump valve of similar material. The tank shall be equipped with a stainless steel immersion type heater capable of raising a solution filled tank to a maximum of 200° F during a 2-hour period, and maintaining a given temperature setting within $\pm 10^\circ$ F. The fixture from which materials being cleaned are suspended shall be electrically insulated from the body of the tank.

2.1.2 Spray Rinse Tank - A tank constructed of No. 300 series stainless steel containing a drain of similar material sufficient in size to quickly empty the tank contents. The tank shall be equipped with a spray system capable of simultaneously rinsing all sides of the printed circuit board.

2.1.3 Etching Tank - A tank constructed of polyethylene, polypropylene or No. 316 stainless steel containing a drain and valve of similar material.

2.1.4 Acid Dip Tank - A tank constructed of polyethylene polypropylene or of mild steel having an internal lining of PVC material and the exterior suitably protected from corrosion. The fixture from which the printed circuit material being cleaned is suspended shall be electrically insulated from the body of the tank.

2.1.5 Deionized Water Dip Tank - A dip rinse tank of polypropylene, polyethylene or of No. 316 stainless steel containing a drain and valve of similar material and either a dam or standpipe type overflow.

2.2 Materials

2.2.1 Cleaner (organic removal) - Neutracleen No. 7, manufactured by Shipley Company, 2300 Washington St., Newton, Mass.

2.2.2 Etch - Ammonium Persulfate, Crystals, Anhydrous

2.2.3 Acid - Hydrochloric Acid - 36.5-38 percent, Reagent, A.C.S.

2.2.4 Deionized Water - Maximum conductivity 50 micromhos, temperature 70-90° F, pressure 60-90 psi.

3. PROCEDURE FOR TANK MAKEUP

3.1 Clean (organic removal) - Mix 12 ounces of Neutracleen No. 7 per 1 gallon of deionized water.

3.2 Etch - Mix 2 pounds of Ammonium Persulfate per 1 gallon of deionized water. The use of warm water after the initial addition of cold water to the tank is recommended in order to rapidly obtain the operating temperature of 70-90° F desired since Ammonium Persulfate upon dissolving tends to produce considerable solution cooling.

3.3 Acid Dip - Add 20 to 30 percent by volume of Hydrochloric Acid to Deionized water to operating volume.

4. PROCEDURE FOR CLEANING RESIST COATED COPPER CLAD FOR PLATING

4.1 Clean (organic removal)

- (a) Immerse board completely into cleaning tank containing a solution of Neutracleen No. 7 that has been raised to desired operating temperature. Immersion time shall vary with solution operating temperature chosen as follows:

130° F -----10-12 minutes

150° F ----- 5-8 minutes

180° F ----- 3-5 minutes

200° F ----- 2-3 minutes

- (b) Rinse board in spray rinse tank for 30 to 45 seconds.

4.2 Etch

- (a) Immerse board completely into etch tank containing a solution of Ammonium Persulfate for approximately 3 seconds.
- (b) Remove board completely from solution and hold in air for approximately 3 seconds.
- (c) Repeat steps (a) and (b) 10 times, or until a uniform pink color appears on the copper surface(s). Immersion time should not exceed 40 seconds total in the solution in order to prevent removal of excessive copper.
- (d) Rinse board in spray rinse tank for 30 to 45 seconds.

4.3 Acid Dip

- (a) Immerse board completely in the hydrochloric acid solution for a continuous period of 30 to 45 seconds.
- (b) Dip and move about in the dip tank for a period of 15 to 20 seconds then transferred within 30 seconds after initiation of the rinsing operation to the plating operation as specified on the appropriate drawing.

5. QUALITY CONTROL

5.1 Control of Chemical Cleaning Solutions

5.1.1 Cleaner - Neutraclean No. 7 - Dump tank when solution will not remove oxides within 1 minute when tank temperature is 150° F or above. Dump tank weekly on a regularly scheduled basis.

5.1.2 Etch - Ammonium Persulfate - Make up new bath daily. Make sure that the bath temperature is at least 70° F. before using. The use of hot water to bring up the temperature is permissible when added after cold water starts the dissolving action of the crystals.

5.1.3 Acid Dip - Hydrochloric Acid - Laboratory analysis at least weekly holding solution concentration between 20 and 30 percent by volume. Dump solution when solution becomes cloudy, milky, or tinged with yellow.

5.2 Additional Optional Method of Control - Since the proof of proper chemical cleaning before electroplating is the ability of the electroplated sub-surface to be completely wetted by molten solder this technique then becomes an excellent method of verifying the chemical cleaning used. It should be noted that when the electroplate is gold that there will appear to be more graininess in the solder coating than when the electroplate is solder. This additional grainy appearance is to be expected with gold plate and should not be used as a criteria for solder coating. The correct criteria for judgment of surface cleanliness is the absence of dewet solder areas or pin holes in the solder after the excess solder is removed in a solder leveling operation.

Where the electroplate applied is not dissolvable (as with gold) or fusible (as with solder) this technique cannot be utilized. Possible electroplates which when applied, that cannot be tested utilizing this technique include Nickel (which will not completely dissolve to the basis metal) and Rhodium which will not react with molten solder.

PROCESS CONTROL AND OPERATING PROCEDURES FOR COPPER PLATING OF SPACE VEHICLE PARTS

1. SCOPE

1.1 Scope - This manufacturing process data covers the necessary precautions and procedures for process control of copper plating of space vehicle parts.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Handling fixtures
- Laboratory instruments titrating and volumetric equipment
- Graduate cylinder (100 ml.)
- Erlenmeyer flask
- Platinum cathode
- Analytical balance
- Beaker (400 ml)
- Stalagmometer

2.2 Materials

- Copper plating bath sample
- Standard thiosulfate solution
- Distilled water
- Potassium iodide

- Ammonium hydroxide
- Silver nitrate (0.1 N)
- Acetic acid (30 percent)
- Potassium hydroxide
- Sulfuric acid (1 N)
- Zinc metal powder
- Hydrochloric acid
- Potassium iodate
- #41 Whatman filter paper
- Nitric acid

3. PROCEDURE

3.1 Solution control and operating conditions

3.1.1 Determine the copper content electrolysis:

- (a) Pipette a 10 ml sample in a 400 ml beaker.
- (b) Add 10 ml of sulfuric and 10 ml of nitric acid, under a hood, and evaporate to dryness, then cool.
- (c) Dissolve salts in 100 mls of warm distilled water and add 1 to 2 ml of nitric acid.
- (d) Weigh platinum cathode. Immerse electrodes in plating solution, leaving upper fourth exposed, and electro deposit the copper on the cathode at 2 to 3 amps for at least 1 hour.
- (e) Add water and immerse for 10 minutes and see if any copper is plated out on upper fourth of cathode. If so, continue electrolysis for 1/2 hour.

(f) If no more copper is plated, wash cathode with water upon removal.

(g) Re-weigh and determine the amount of copper.

(h) Calculate:

$$\begin{aligned}\text{Grams of Cu} \times 12.4 &= \text{oz/gal Cu (metal)} \\ \text{oz/gal Cu (metal)} \times 3.9 &= \text{oz/gal Coppralyte (K)} \\ \text{oz/gal Cu} \times 3.7 &= \text{oz/gal coppralyte (Na)}\end{aligned}$$

3. 1. 2 Determine the free potassium cyanide:

(a) Pipette 10 ml sample in a 250 ml Erlenmeyer flask.

(b) Add 100 ml distilled water, 5 ml of 10 percent potassium iodide and 15 ml of concentrated ammonium hydroxide.

(c) Titrate with standard 0.1 N silver nitrate to a milky color which remains for 1 minute.

(d) Calculate:

$$\begin{aligned}\text{ml of } 0.1 \text{ N AgNO}_3 \times 0.174 &= \\ \text{oz/gal KCN (Free)} & \\ 0.174 = 2 \frac{(\text{eq. wt. of KCN})}{10} &\times 0.134\end{aligned}$$

3. 1. 3 Determine the potassium hydroxide

(a) Pipette 10 ml sample into a 250 ml Erlenmeyer flask.

(b) Add 6 to 8 drops La Matte Sulfo-orange indicator.

(c) Titrate with 1 N sulfuric acid until deep orange turns to greenish yellow.

(d) Calculate:

$$\text{mls of 1 N H}_2\text{SO}_4 \times 0.752 = \text{oz/gal KOH}$$

$$0.752 = \frac{(\text{eq. wt. of KOH})}{10} \quad 0.134$$

3. 1. 4 Control the use of addition agent 1085 by use of a Stalagmometer at 25° C.

Standard Solution	Drops Between Lines
No mls/gal addition agent	42.5
1 ml	48.0
2 ml	54.8
3 ml	59.0
4 ml	64.0
5 ml	65.0
7.5 ml	67.0
10 ml	75.0
15 ml	87.0

To determine concentration of unknown, pipette 5 ml in beaker and add 15 ml cutting solution and count drops between lines.

Calculations: Get number of drops of sample it takes for liquid level to go from top line to bottom line on stalagmometer and then use above table to determine the concentration of the 1085 addition agent in ml/gal.

3. 1. 5 Determine the sodium thiocyanate content:

(a) Place 5 grams of zinc metal powder in a 250 ml widemouth Erlenmeyer flask.

(b) Pipette 2 ml of copper plating solution into the flask.

(c) Add 25 ml of caustic solution and digest for 5 minutes on a hot plate with occasional shaking.

(d) Allow the sample to cool and add 50 ml of water and 10 ml of HCl under a hood.

(e) Filter through a No. 41 Whatman filter paper or its equivalent, and wash with water, catching the filtrate in a 500 ml Erlenmeyer flask.

(f) To the filtrate, add 20 ml of HCl and dilute to about 200 ml. Cool the sample to room temperature.

(g) Pipette exactly 50 ml of $\frac{N}{10}$ KIO₃ solution into the sample and mix.

(h) Add approximately 1.5 grams of KI crystals and back titrate with Na₂ S₂ O₃ solution until the resulting blue color is just discharged. Add 2 to 3 drops of starch solution and continue the titration until the resulting blue color is just discharged. Record the total titration.

(i) Calculate: $5.0 - (\text{ml of Na}_2 \text{S}_2 \text{O}_3 \times N) \times 0.905 = \text{oz/gal Na CNS}$
KIO₃ N/10 dissolve exactly 3.5670 grams of C. P. KIO₃ in water and dilute to 1 liter in a volumetric flask.

3.1.6 Make up sodium hydroxide solution.

(a) Dissolve 400 grams of sodium hydroxide pellets in water; cool and dilute to 1 liter.

3.1.7 Determine the sodium or potassium carbonate content:

(a) Pipette 5 ml of plating solution into a 250 ml widemouth Erlenmeyer flask.

(b) Add 100 ml of water; heat nearly to boiling and add 30 ml of clear 10 percent barium chloride solution.

(c) Stopper the flask and allow the precipitate to settle.

(d) Filter through No. 41 Whatman paper or equivalent, and wash the barium chloride solution.

(e) Carefully return the filter paper containing the precipitate to the original flask, add 100 ml of water, then heat to 80°C.

(f) Add 5 drops of the indicator and titrate while hot with N/10 hydrochloric acid to a greenish-purple endpoint. Stopper the flask and shake vigorously near the apparent endpoint until the greenish-purple color is permanent. Record the titration.

(g) Calculate:

$$\text{ml N/10 hydrochloric acid} \times 0.142 =$$

$$\text{oz/gal Na}_2\text{CO}_3 \quad \frac{0.134 (\text{ml} \times 0.1 \text{ N HCl}) (\text{eq. Na}_2\text{CO}_3)}{5 \text{ ml sample}}$$

(h) Carbonate Removal:

(1) Carbonate may be precipitated by cooling solution to 26° F.

(2) Carbonate may be removed by adding either barium hydroxide or calcium hydroxide.

1 oz/gal Na₂CO₃ requires 3 oz/gal Ba (OH)₂ 8H₂O; forms 0.8 oz/gal NaOH

1 oz/gal K₂CO₃ requires 2.3 oz/gal Ba (OH)₂ 8H₂O; forms 0.8 oz/gal KOH

1 oz/gal Na₂CO₃ requires 0.7 oz/gal Ca (OH)₂; forms 0.8 oz/gal NaOH

1 oz/gal K₂CO₃ requires 0.54 oz/gal Ca (OH)₂; forms 0.8 oz/gal KOH

4. CONTROL

4.1 Determine the copper content by electrolysis, free potassium cyanide, potassium hydroxide and carbonate content once per week.

4.2 Insure that beakers, pipettes and all laboratory instruments are maintained in a clean condition.

4.3 Check normality of solutions against known standards once per week or as often as solutions are made up.

MANUFACTURING PROCESS DATA FOR CLEANING OF TARNISHED BIFURCATED SILVER TERMINAL BOARDS

1. SCOPE

1.1 Scope - This manufacturing process data covers the cleaning and package preservation of bifurcated silver terminals.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Thermal impulse sealer
- Process and rinse tanks
- Temperature controls
- Safety equipment (goggles, gloves, etc.)

2.2 Materials

- MacDermid Electrocleaner, Anodex NP No. 2 or Oakite 90
- Demineralized water
- Sodium cyanide
- Polyethylene bags
- Mothballs

3. PROCEDURE

3.1 Alkaline clean for 10 minutes in MacDermid Electrocleaner, Anodex NP No. 2 or Oakite 90 at a concentration of 5 to 7 ounces per gallon and a temperature of 160 to 190°F.

- 3. 2 Rinse in warm demineralized water for approximately 1 minute.
- 3. 3 Clean in sodium cyanide solution for 15 to 20 seconds at a concentration of 10 to 15 ounces per gallon and at ambient temperature.

CAUTION: Handle Cyanide Solution with care.

- 3. 4 Rinse in cold demineralized water for 3 minutes minimum.
- 3. 5 Rinse in hot demineralized water for 1/2 to 1 minute (160° F maximum).
- 3. 6 Dry at 160° F maximum, vacuum drying can be used.

CAUTION: Remove water prior to packaging.

- 3. 7 Package and seal in polyethylene bags with four mothballs per bag.

4. CONTROL

- 4. 1 Extreme care must be exercised while working with cyanide. Do not allow solution to contact eyes, mouth or any exposed cut or scratch on the skin; thoroughly wash hands after processing terminals.
- 4. 2 All water entrapped in terminal sleeve must be removed prior to packaging.

MANUFACTURING PROCESS DATA FOR
CLEANING OF ATM SILVER PLATED COMPONENTS

1. SCOPE

1.1 Scope - This Manufacturing Procedure covers the final cleaning and packaging of Silver Plated ATM components.

2. APPLICABLE DOCUMENTS

2.1 Federal

Federal Standard 209A - "Clean Room and Work Station Requirements, Controlled Environments".

George C. Marshall Space Flight Center

MSFC-STD-246A - "Design and Operational Criteria of Controlled Environmental Areas".

MSFC-SPEC-164 "Cleanliness of Components for Use in Oxygen, Fuel and Pneumatic".

MSFC-PROC-151 "Contamination Control and Environmental Protection of Space Vehicles and Associated Equipment Procedure for".

MSFC-50M02412 "ATM Cleanliness Specification".

MSFC-30M14500 "Apollo Telescope Mount Assembly".

3.0 REQUIREMENTS

3.1 Facilities

- Bldg. 4705 - Tube cleaning
- Bldg. 4707 - Clean room

- Bldg. 4755 - 30 000 and 100 000 Class Clean Rooms

3.2 Environmental Cleanliness

- 30 000 Class Clean Room - 30 000 particles per cubic foot greater than 0.5 microns per cubic foot and 215 particles greater than 5 microns per cubic foot.
- 100 000 Class Clean Room - 100 000 particles greater than 0.5 microns per cubic foot and 700 particles greater than 5.0 microns per cubic foot.
- Relative Humidity shall not exceed 50 percent at 72° F.

3.3 Equipment

- Vacuum Oven
- Heat Sealer
- Ultrasonic Equipment

3.4 Chemicals and Materials

- Acetone
- Triflouro, Trichloro Ethane, (Freon, PCA).
- MEK
- Trichlorethylene Types I & II
- Dowell F-33 Detergent
- Nylon-6 Film, 2 to 4 mils
- Nylon Gloves
- Nylon Wipe Cloths
- Polyethylene Film, 4 to 6 mils

3.5 Other Requirements

3.5.1 All components shall be double bag sealed with inner bag being Nylon-6 and outer bag being Polyethylene.

3.5.2 Cleanliness- The final cleaning of all components shall meet the requirements of MSFC-SPEC-164.

3.5.3 All components shall be handled with Nylon gloves.

4. CLEANING PROCEDURE

4.1 Precleaning - Parts shall be precleaned with a Nylon cloth moistened with Acetone to remove oil, grease, dirt or ink.

4.2 Degrease - Parts shall be vapor degreased in trichloroethylene.

4.3 Dry - Parts shall be statically air dried with missile grade air at temperature of 150 to 180° F.

4.4 Ultrasonic Cleaning - Parts shall be ultrasonically cleaned in a 0.5 percent solution of Dowell F-33 detergent at 130° F ($\pm 10^\circ$) for 3 to 5 minutes.

4.5 Rinse - Parts shall be rinsed in hot demineralized water at 120° F ($\pm 10^\circ$) until suds free.

4.6 Dry - Parts shall be dried in vacuum oven at 25 to 27 inches mercury at 110° F ($\pm 10^\circ$) for 1 hour.

4.7 Final Cleaning - Parts shall be cleaned with pre-filtered Freon, PCA.

4.8 Cleanliness - Particulate matter and NVR shall meet the requirements of MSFC-SPEC-164.

4.9 Dry - Parts shall be dried in a vacuum oven at 25 to 27 inches of mercury at 110° F ($\pm 10^\circ$) until dry.

4.10 Package - Parts shall be double bag sealed with inner bag being Nylon-6 and outer bag being Polyethylene.

5. QUALITY ASSURANCE

5.1 Inspection Points

5.1.1 See paragraph 3.2.

5.1.2 See paragraph 4.8.

MANUFACTURE OF SINGLE AND DOUBLE SIDED GOLD-PLATED PRINTED CIRCUIT BOARDS

1. SCOPE

This Manufacturing Process Data (MPD) covers the manufacture of single and double sided gold-plated printed circuit boards.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Copy camera and arc lamps
- Photographic film developing pans (develop, stop, and fix)
- Film wash basin
- Film drying cabinet
- Contact printer
- Drying oven
- Power driven scrub brush
- KPR spray gun and dip tank
- Vapor degreaser (Trichloroethylene)
- Etching machine (ferric chloride)
- Plating console
- Shadowgraph
- Tape control multi-spindle drill
- Light table

2.2 Material

- Pumice (Freemont No. 238)
- Entek number 55
- Kodak photo resist
- Hydrochloric acid
- Sulfuric acid
- Trichloroethylene
- Ammonium persulfate
- Cold-Strip PR-491 (Allied Finishing Specialties Co.)
- DuPont number 21D developer
- Acedic Acid (stop)
- DuPont number 18F fixer
- Gold plating solution
- Copper-clad laminate (per MIL-P-13949)
- Polyethylene bags
- Stable plastic film (per L-P-00517 or L-P-00519)
- Black opaque drawing ink (per TT-I-00526)
- Oxalic acid
- Layout tape and assorted templates of opaque, black, matte-surface, adhesive back, pressure sensitive material

3. PROCEDURE

3.1 Art Work Preparation

3.1.1 Prepare master art work on dimensionally stable plastic film not less than 0.004-inch thick. Art work may be prepared using either opaque pressure sensitive tape and templates or black opaque drawing ink. Taped or hand drawn art work shall be 1-to-1 scale unless tolerance or equipment limitations dictates otherwise. Machine drawn art work may be 1-to-1 scale. Art work shall provide well defined registration marks to control front to back registration on double sided boards. Marks shall be accurately located and spaced such that when the distance between them is photographically reduced to 4 inches, the resultant transparency is correctly scaled.

3.1.2 Art work layout shall provide for a removable tab adjacent to circuit pattern for quality control purposes. The tab should have circuitry representative of the circuits on the board.

3.1.3 Compute area of circuitry and provide this information to the plater for setting current levels during plating.

3.2 Chronar Preparation - This part of MPD will be supplied later when this part of facility is operable.

3.3 Transparency Preparation

3.3.1 The true scale transparency used for printing circuit image on board is made by photographing, with the copy camera, the art work master. (MSFC-STD-154 calls this the "Master Pattern".) (The Chronar will be used to produce transparency when Chronar process is implemented.) Care should be exercised to maintain flatness of master during photographing and to obtain sharp focus and well defined image. Carefully measure the 4-inch distance between registration marks on reduced image to assure an accurately scaled transparency. Expose film for 3 seconds at a lense iris opening of F22.

3.3.2 Develop film for approximately 2.5 minutes in Du Pont 21D developer. Mix A and B parts 1-to-1. Mix fresh daily. Stop development in 28 percent acidic acid dilute with water 8 parts water to 1 part acid. Fix film in DuPont 18F. Discard fix solution and draw fresh material daily. After fixing, wash prints thoroughly in film wash basin and dry completely prior to use.

3.4 Board Manufacture

3. 4. 1 Cut boards to rough size allowing material for final trim and an area to connect electroplating contacts.

3. 4. 2 Scrub boards thoroughly using power brush, pumice and water. Remove all visible corrosion and contamination. Wash in water after scrubbing. Boards must be swabbed during washing to assure removal of all pumice, etc. Rinse in water and blow dry with oil free (missile grade) air. Avoid touching boards with bare hands after this cleaning.

3. 4. 3 If boards are not processed immediately after the above washing, dip boards in Entek 55, drain and blow dry with oil free air. Before further processing of boards, remove Entek in HCL dip. Rinse thoroughly with water to remove all Entek and HCL residue and blow dry with oil free air.

3. 4. 4 Apply KPR. This may be applied either by spraying or dipping. If sprayed, spray twice, once with vertical strokes and once with horizontal strokes or spray with vertical strokes and rotate board 90 degrees and allow the material to flow across spray strokes. If dipped, allow boards to drain standing on edge to assure uniform coating. Allow KPR to air dry until it is not tacky, then oven dry for 15 minutes at 205° F.

3. 4. 5 Expose board using small contact printer. To register transparencies for double-sided boards position transparencies on opposite sides of a piece of glass or transparent plastic of approximately the same thickness as the board. Secure transparencies along their edges to a strip of the glass or plastic. Remove glass or plastic and insert board blank taking care to prevent movement of one transparency with respect to the other. Expose one side using contact printer. Invert and expose other side taking care not to permit board or transparencies to move with respect to each other. Inspect transparencies carefully prior to each use to assure that they have no damage that would degrade finished printed circuit boards.

3. 4. 6 Develop boards in trichloroethylene vapor degreaser. Development can be done using commercial developing solution. When these materials are used, follow the manufacturer's instructions.

3. 4. 7 Bake board at 200° F for 5 minutes to harden resist softened by developing process.

3. 4. 8 Anodic clean in alkaline for 5 to 10 minutes at solution temperature of 110 to 130° F (6-8 ounces per gallon).

- 3. 4. 9 Rinse in water to completely remove alkaline solution.
 - 3. 4. 10 Preplating etch for 15 to 60 seconds in ammonium persulfate solution, (5 to 7 ounces per gallon), as required to remove surface oxidation.
 - 3. 4. 11 Wash thoroughly in warm water spray rinse. Check for complete removal of ammonium persulfate by checking for water breaks on wet surface.
 - 3. 4. 12 Wash in 27 to 33 percent hydrochloric acid solution or in 20 to 30 percent sulfuric acid solution.
 - 3. 4. 13 Rinse thoroughly (at least twice).
 - 3. 4. 14 Gold strike to 5 to 10 seconds at 6 volts and 65 to 95°F (0.05 to 0.2 troy ounces per gallon).
 - 3. 4. 15 Gold plate at 10 to 15 amperes per square-foot of surface until 100 to 200 millionth of gold has been deposited on surface.
- NOTE: Plating amperes may be obtained by multiplying the total area in square feet of the circuit being plated by 12. The plating time in minutes may be obtained by multiplying the total area being plated by the amperes and dividing the results into 55.43 (0.7 to 2.0 troy ounces/gallon).
- 3. 4. 16 Rinse in water and blow dry with oil free air.
 - 3. 4. 17 Strip KPR
 - 3. 4. 18 Etch boards in ferric chloride. Observe etching process to assure complete etch with minimum of over etch.
 - 3. 4. 19 Rinse thoroughly in water.
 - 3. 4. 20 Rinse in 4 to 6 percent solution, by volume, of hydrochloric or oxalic acid.
 - 3. 4. 21 Rinse thoroughly in warm water spray. This is a critical step and care should be taken to assure complete removal of all reagents.
 - 3. 4. 22 Blow off water with oil free air and air dry boards.

3. 4. 23 Carefully inspect boards for conformance to the requirements of paragraphs 5. 3. 3 through 5. 3. 6 of MSFC-STD-154A.

3. 4. 24 Store finish boards in unsealed polyethylene bag between further process steps until boards are installed in next assembly.

3. 5 Drilling and Trimming

3. 5. 1 Read coordinates of all drilled holes using shadowgraph.

3. 5. 2 Prepare tape for tape control printed circuit board drill machine from coordinates obtain on shadowgraph.

3. 5. 3 Drill index holes using jigborer.

3. 5. 4 Drill circuit holes on printed circuit drill.

3. 5. 5 Trim board to final size. Cut off control tab and return to printed circuit shop for testing and/or delivery to Quality.

NOTE: This method of prepared drill control tape is an interim procedure which will be replaced when other means become available.

3. 5. 6 Clean board in vapor degreaser to remove contaminations from drilling operation.

3. 5. 7 Wash in warm water and blow dry in oil free air.

3. 5. 8 Deliver board to Quality for acceptance for intended use. After acceptance, store board in polyethylene bags.

3. 6 Process Controls

3. 6. 1 Solution shall be analyzed prior to manufacturing flight printed circuit board if more than 30 days has elapsed since last analysis. Solution shall be analyzed at 250 board intervals until experience data is available to accurately indicate analysis periods.

CLEANING AND GOLD PLATING ALUMINUM
ALLOYS SPECIFIC FOR M512 SPACE EXPERIMENTS

1. SCOPE

This manufacturing procedure covers the requirements for Gold Plating Aluminum Components.

2. APPLICABLE DOCUMENTS

2.1 Federal

2.1.1 Federal Test Method Std. No. 151 - metals

2.2 ASTM

2.2.1 ASTM-E-216-63T - Measuring Coating Thickness by Magnetic or Electromagnetic methods.

3. REQUIREMENTS

3.1 Chemicals

- Acetone
- Trichloroethylene Type II
- Nitric Acid
- Alumon (Zincating Salts)
- Sodium Hydroxide
- Plating Solutions
- Gold plating Solution (Sel Rex 125)

- Nickel Plating Solution (watts)
- Copper Plating Solution
- Zincating Solution

3.2 Materials

- Plastic Bags
- Soft Packing Tissue
- Micrometer
- Dermatron
- Gloves, Rubber
- Gloves, Collar or Nylon
- Test Specimen - shall be of the same base metal used in production with dimension 4 inches x 1 inch x 0.080 inch.

3.3 Equipment

- Large Oven
- pH Meter
- Dermatron

3.4 Physical Test Requirement

3.4.1 Thickness - The thickness shall be specified by the drawing or work order and determined from control sample and/or part.

3.4.2 Adhesion - shall pass Adhesion Test specified by paragraph 3.7.2.

3.5 Bath Maintenance

3.5.1 Complete chemical analysis every third day during production.

3.5.2 Replenishing Bath - Bath shall be replenished as analysis dictates.

3.6 General Requirements

3.6.1 Bake - After gold plating, specimen shall be baked at $375 \pm 25^{\circ}\text{F}$ for 3 hours.

3.7 Detail Requirements

3.7.1 Thickness of Coating

3.7.1.1 Copper - Unless otherwise specified, the minimum thickness of copper coating shall be 0.0001 inch.

3.7.1.2 Nickel - Unless otherwise specified, the minimum thickness of watt nickel shall be 0.0001 inch.

3.7.1.3 Gold - Unless otherwise specified, the minimum thickness of the Sel-Rex 125 gold shall be 0.0001 inch.

3.7.2 Adhesion - The adhesion shall be such that when examined at a magnification of four diameters, the gold coating shall not show separation at the interface of the base metal. The formation of cracks in the base metal or gold coating, which does not result in flaking, peeling, or blistering, shall not be considered as non-conformance to this requirement.

3.7.3 Workmanship - The gold coating shall be smooth, adherent, and free from visible blisters, pits, nodules, porosity or other defects. Slight discoloration resulting from heat treatment shall not be a cause for rejection.

3.7.4 Control Sample - shall be run parallel with production parts.

4. PROCEDURE

4.1 Precleaning - The parts shall be precleaned with Acetone or mechanical cleaning to remove soil, grease or inks.

4.2 Etch - Parts shall be flash etched in a 1 oz. \pm gal. caustic solution at $200 \pm 10^{\circ}\text{F}$ for 10 to 60 seconds.

4. 2. 1 Optional - The parts shall be etched in a 40 ± 5 percent by weight solution of Sulfuric Acid and 5 ± 1 percent by weight solution of Sodium Dichromate for 1 to 3 minutes.
4. 3 Rinse - The parts shall be rinsed to remove the caustic solution.
4. 4 Desmut - The parts shall be immersed in a fresh, clean 50 percent by volume of Nitric Acid solution until desmuted.
4. 5 Rinse - The parts shall be rinsed for a minimum of 2 minutes in cold water.
4. 6 Zincate Process
4. 6. 1 Zincating - The parts shall be immersed in concentrated alumon solution for 10 to 60 seconds depending on the alloy.
4. 6. 2 Rinse - The parts shall be rinsed in cold water for a minimum of two minutes.
4. 6. 3 Pickle - The zincated parts shall be immersed in a fresh 50 percent by volume of Nitric Acid solution for 20 to 60 seconds.
4. 6. 4 Rinse - The parts shall be rinsed in cold water for a minimum of 2 minutes.
4. 6. 5 Re-Zincate Process - Refer to paragraph 4. 6. 1, if the parts require additional zincating.
4. 6. 6 Rinse - The parts shall be rinsed in DI water.
4. 7 Copper Plating - The parts shall be immersed in the copper plating solution with the current on and raised for a few seconds to flash coat the parts. Follow this process by reducing the current density for copper plating the parts.
4. 7. 1 Thickness - A thickness of 0.0001 to 0.001 ml of copper shall be attained.
4. 7. 2 Rinse - The copper plated parts shall be rinsed in cold rinse.

4.7.3 Inspect - The plater shall visually inspect the part for blisters or other defects.

4.7.3.1 Blisters - If blisters occur, the parts shall be stripped in 50 percent by volume of Nitric Acid and then processed per paragraphs 4.2 to

4.7.3.

4.8 Nickel Plating - Required. The parts shall show no water breaks oxidation before immersing in the nickel plate solution. Process the parts in the nickel plate bath at the proper current density.

4.9 Rinse - The parts shall be rinsed in cold water.

4.10 Gold Plate - The parts shall be immersed in the gold plating solution at the proper current density.

4.10.1 Thickness - The thickness shall be specified by the work order or drawing number.

4.11 Rinse - The parts shall be rinsed in cold water.

4.12 Dry - The parts shall be dried at ambient temperature.

4.13 Heat Treat

4.13.1 Specimen - The specimen shall be heat treated at 375° F (±25°) for 3 hours. Allow parts to cool to ambient temperature.

NOTE: If the specimen fails, the planner or designer needs to be called to determine if the part needs the above heat treatment.

4.13.2 Optional - If required the parts shall be heat treated at 375° F (±25°) for 3 hours. If blisters or other defects occur, the parts shall be stripped and re-processed per paragraph 4.1 to 4.7.3.

4.14 Discoloration - Any discoloration present shall be removed with fine steel wool or stainless steel brushes.

4.15 Package - The parts shall be packaged by wrapping with soft materials and placed in poethylene bags.

5. QUALITY ASSURANCE

5.1 Inspection Points

5.1.1 Visual Inspection - Parts shall be visually inspected for workmanship - Reference paragraph 3.7.3.

5.1.2 Thickness - reference paragraph 3.7.1.

5.1.3 Adhesion - reference paragraph 3.7.3

5.1.4 Bake - reference paragraph 3.6.1 for conformance with paragraph

MANUFACTURING SPECIFICATION FOR THE
FABRICATING OF ADHESIVELY BONDED HONEYCOMB STRUCTURES
USING CORE AND FACE SHEETS

1. SCOPE

1.1 Scope - This Manufacturing Process covers the Process Engineering Laboratory requirements for the fabrication of adhesively bonded composite structures from metallic and non-metallic skins, and honeycomb core, intended for use in space vehicle applications. This document is a revision of M-ME-MPROC-005. 5B, dated Oct. 1963.

1.2 Applicability - The provisions of this process are applicable to all honeycomb composite structures fabricated by or for the Process Engineering Laboratory for service within the temperature range as specified by design drawings.

1.3 Restrictions - No other materials or processes may be used as alternates or equivalents to those required by this document without the joint approval of S&E-ME-D and S&E-ME-M.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents, of the issue in effect on the date of use of this process, form a part of this process to the extent indicated herein.

2.1.1 Specifications

Federal

QQ-A-255A (1)	Aluminum Alloy, Plates and Sheets, Alclad 2014 (R301, Clad 148)
QQ-A-283A	Aluminum Alloy, Plate and Sheet, 7075
QQ-A-287A	Aluminum Alloy, Plate and Sheet, Alclad 7075

CCC-C-440(1)	Cloth, Cotton Cheesecloth
UU-P-268	Paper, Kraft, Untreated, Wrapping
P-P-101	Paper, Abrasive, Artificial, Water-proof
NPC 200-3	Inspection System Provisions for Suppliers of Space Materials, Parts, Components, and Service
F-29-60T	American Society for Testing & Materials
TTM-261	Methyl-Ethyl-Ketone

Military

JAN-T-171(2)	Toluene
MIL-G-3866A	Gloves, Cloth, Cotton, Knitted, Lightweight
MIL-C-7438D(2)	Core Material, Aluminum, for Sandwich Construction
MIL-M-13999	Methyl-Ethyl-Ketone, Analyzed Reagent
MIL-P-14591A	Plastic Film, Nonrigid, Transparent
MIL-P-17667A(1)	Paper, Wrapping, Chemically Neutral
MIL-A-19842B(1)	Aluminum Alloy Plates and Sheets, 5456
MIL-A-25463(1)	Adhesive, Metallic Structural Sandwich Construction
MIL-P-6889A	Zinc Chromate Primer
JAN-T-85	Zinc Chromate Primer Thinner
MIL-A-8623	Adhesives, Epoxy Resin, Metal-to-Metal Structural Bonding

MIL-P-8073	Core Material, Plastic Honeycomb, Laminated Glass Fabric Base, for Air- craft Structural Application
MIL-S-11161	Sodium Dichromate, Reagent Grade
MIL-C-8073A	Core Material, Plastic Honeycomb

George C. Marshall Space Flight Center

MSFC SPEC 137A (AMDT 1)	Aluminum Alloy, Plate and Sheet, Specification for
MS 150.0	Manufacturing Specification for Vapor Degreasing of Metallic Surfaces
M-ME-MPROC 005.5B-3	Cleaning for Adhesive Bonding
MMM-A-132	Adhesives, Heat Resistant, Air Frame Structural, Metal-to-Metal
MSFC-Mel	Composite Structures Manual (for internal use only) or Manufacturer's Data Sheets (for Contractors)

Federal Standards

151a	Metals: Test Methods
175	Adhesives: Methods of Testing

2.2 Other Publications - The following documents form a part of this process. Unless otherwise indicated, the issue in effect on the date of issuance of this process shall apply.

Society of Automotive Engineers, Inc.

AMS 4028	Aluminum Alloy Plate and Sheet, 4.5 Cu - 0.8 Si - 0.8 Mn - 0.5 Mg, 2014-0
AMS 4029	Aluminum Alloy Sheet and Plate, 4.5 Cu - 0.8 Si - 0.8 Mn - 0.5 Mg, 2014-T6

3. GENERAL REQUIREMENTS

3.1 Deviations - No deviations from the requirements of this procedure or those of the detail procedures will be permitted without written approval of S&E-ME-M and S&E-ME-D.

3.2 : Cleanliness - Cleanliness is a key requirement of adhesive bonding and refers to the condition of the structural components from the time they leave the cleaning tank until they are removed from the autoclave as bonded assemblies. The cleaned parts must be free of any contamination prior to application of the primer and adhesive. All cleaning procedures are specified in M-ME-MPROC-005.5B-3 - Cleaning for adhesive bonding.

3.3 Handling and Lay-Up - Handling and lay-up must be planned and performed to avoid contamination of faying surfaces.

3.4 Tooling and Equipment - All tooling and equipment used in the bonding processes specified herein and in the detail specification are required to possess the inherent capability to produce bonded joints that will meet or exceed specified requirements.

3.4.1 Fixtures or jigs (as applicable) - Provide a fixture or jig to hold details in firm contact in all bond areas and in proper alignment with each other during splice cures.

3.4.2 Final assembly bonding tools - The final assembly bonding tools are provided with means for locating and indexing core and other detail parts or subassemblies. All tooling areas that contact adhesive are coated with an approved parting agent that will not contaminate the bond.

3.4.3 Qualification - Prior to use in production, each set of tools or piece of equipment is to be qualified by first bonding a process test specimen. This specimen will be used to determine the quality of the bond produced by the tools and equipment. The first production part from a new tool or piece of equipment or any tool or piece of equipment subjected to any major rework should be subjected to such tests as deemed necessary to assure adequacy of tooling and equipment and bonding procedures specified for the part. Each

investigation of questionable bonding or evaluation of bonding processes should include a check of related tools, equipment, and processes.

3.4.4 Equipment

- Vapor degreasing equipment - Trichloroethylene type.
- Spray gun - DeVilbiss gun, equipped with fluid needle MBC-444FX, fluid tip AV-15-FX, and air cap number 36, or equal.
- Autoclave - An autoclave capable of handling the parts to be bonded is required for use in bonding of parts.
- Pressure bags - Materials used must provide a flexible impermeable barrier between the pressure medium and the part being bonded. A suitable bleeder material should be used between the part and the pressure bag.
- Vacuum pump - A vacuum pump of suitable capacity is required for use in evacuating the sealed assembly.

3.5 Safety

3.5.1 All Standard shop safety precautions are applicable during all stages of fabrication, from cleaning, machining and forming, to the final bonding.

3.6 Cleaning

3.6.1 All cleaning procedures for honeycomb bonded structures are contained in specification M-ME-MPROC 005.5B-3, Cleaning for Adhesive Bonding.

4. MATERIALS

4.1 Adhesives - The scope of this procedure covers all metallic and non-metallic materials, face sheets and cores that are used to fabricate adhesively bonded honeycomb structures in the Process Engineering Laboratory.

4.1.1 Specification - The adhesives used in the production of bonded metal-to-core or plastic-to-core composite structures must meet all the requirements of MIL-A-25463, or as approved by the project engineer, for the type adhesive specified on the detail drawing.

4.1.2 Certification of adhesives - The vendor supplies documents containing the following information:

- Vendor's adhesive batch* number
- Date of manufacture
- Test values obtained on all tests performed to certify the adhesives
- Date shipped from vendor
- Adhesive storage requirements
- Recommended shelf life at a specified maximum storage temperature

*A batch is defined as that quantity of material which has been manufactured at one time or subjected to some chemical or physical mixing process intended to make the final product homogenous.

4.1.3 Control record - Upon receipt of a new batch of adhesive from the manufacturer, a record of that batch must be originated and maintained until the batch is entirely used up or discarded. This record should contain the following information:

- Manufacturer's batch number
- Purchaser's lot number
- Date of manufacture
- Expiration date of manufacturer's guaranteed shelf life
- Date of acceptance sampling
- Date of acceptance testing
- All acceptance test value
- Storage temperature range (minimum, maximum)
- Scheduled reinspection date (during storage)
- Date and time into and out of refrigerator

- All reinspection dates and test values
- Date of evaluation for release for production use
- All production evaluation test values
- Dates of issue for production

4.1.4 Acceptance testing - Within 72 hours after receipt of each new batch of adhesive, expose the roll or container to room temperature ($70^{\circ} \pm 5^{\circ}\text{F}$) until moisture condensation ceases. Remove a sample from this roll or container and use to bond test specimens. The adhesive material should then be sealed and returned to storage as soon as possible. The specimens are then subjected to the tests outlined in the manufacturer's data sheet, or as defined by S&E-ME-MM.

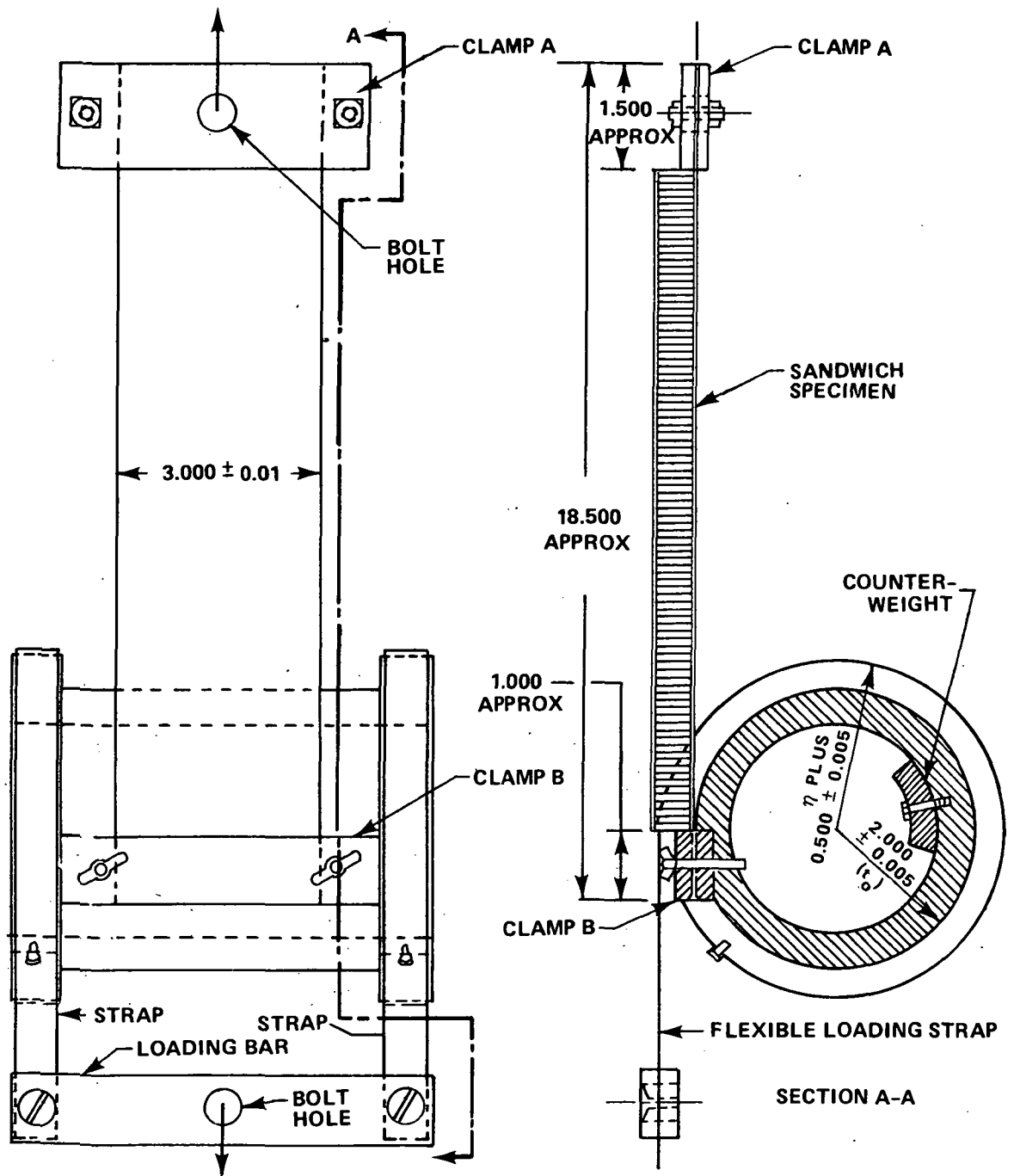
4.1.5 Periodic recertification - Periodic recertification should be performed on all adhesives that have not been released for production use within the guaranteed shelf-life. Periodic recertification consists of subjecting samples to the tests specified in the manufacturer's data sheet, or as defined by S&E-ME-MM for the particular adhesive.

4.1.6 Production evaluation - Adhesive used in the production bonding of composite structures must be preproduction qualification tested as specified in the manufacturer's data sheet or as defined by S&E-ME-MM within 168 hours preceding use. Production evaluation tests processed with the parts consist of the following tests and must meet the minimum requirements specified in the manufacturer's data sheet, or as defined by S&E-ME-MM for the specific adhesive:

4.1.6.1 Normal temperature tensile shear test - For production parts, a minimum of one specimen is made per platen load and tested in accordance with the requirements of MMM-A-132, the manufacturer's data sheet or as defined by S&E-ME-MM. The specimen goes to the Quality Control Group.

4.1.6.2 Metal-to-core climbing drum peel test - Normal temperature sandwich peel tests are to be conducted in accordance with the requirements of MIL-A-25463, the manufacturer's data sheet or as defined by S&E-ME-MM. The test setup is shown in Figure 9-1. A minimum of two specimens must be prepared and tested.

4.2 Honeycomb core material - Honeycomb core material used in the bonding of honeycomb sandwich structures must meet all the requirements of MIL-C-7438D, unless approved by S&E-ME-M and S&E-ME-D.



DIMENSIONS IN INCHES. NOT COMPLETELY DIMENSIONED.
 r_i = RADIUS OF DRUM TO MID-DEPTH OF SPECIMEN FACING.
 r_o = RADIUS TO MID-DEPTH OF STRAP.

Figure 9-1. Core-to-skin peel test apparatus.

4.2.1 Handling and storage of honeycomb core material - Handling of honeycomb core material should be kept to an absolute minimum. When it is handled, boxes, racks, wax-free paper, or supports are required to prevent core material damage or contamination.

5. BONDING PROCEDURE FOR HONEYCOMB STRUCTURES

5.1 Precleaning requirements

5.1.1 Contact surfaces - Contact surfaces of all parts must be free from burrs, dimples, scratches, steel stamped part numbers, or other surface defects. Consult M-ME-PROC 005.5B-3 on cleaning for adhesive bonding.

5.1.2 Edge chamfer - The edge chamfer or radius resulting from the removal of burrs should not exceed 0.010 inch.

5.1.3 Forming dies - Lubricate forming dies used to form parts for adhesive bonding with a compatible material containing no silicones.

5.1.4 Identification - Identify formed parts with the assembly of which they are a part.

5.1.5 Prefitting - Prefit all surfaces to be joined by adhesive bonding so that only a minimum pressure is required to give contact with the surface being joined.

5.1.6 Preparation of honeycomb core

5.1.6.1 Machining of honeycomb core - Machine all honeycomb core, where possible, before vapor degreasing.

A. Chuckling - Prepare the honeycomb core for chucking by a suitable method, such as:

- (1) Bond skins to the core with adhesives so that vacuum chucking may be used to hold the core provided all core and metal surfaces contaminated by the adhesive are removed by subsequent machining; or
- (2) Fill the core cells with water, and freeze sufficiently to hold the core in position while machining; or

- (3) Fill the core cells with Glycol-E-9000, or an approved equal having a softening point of 210° F maximum.

B. Machining - Machining of core details and subassemblies containing honeycomb core must be accomplished without the use of silicones, lubricants, or liquid coolants on the core or the cutting tools, if possible.

C. Tolerances - Unless otherwise specified, the following tolerances apply to machining of honeycomb core:

- (1) Hold the depth of all stepped cuts machined into the core for doublers to the nominal doubler thickness plus an allowance for the bond-line thickness.
- (2) Hold round holes drilled through the cell walls for foamed-in-place fittings to the nominal fitting diameter +0.060, - 0.000 inch.
- (3) The width of all slots cut into the core for flat blade extrusion fittings are to be held to the nominal fitting dimensions +0.030, -0.000 inch.
- (4) Variations of the core surface not related to contour (steps, dishing, surface waves, etc.) must not exceed +0.002 inch variation in 1.0 inch of length or +0.005 inch in 12.0 inches of length.
- (5) When core is machined to match the contour of a solid metal component, all allowance for difference in bond thickness is required. For metal-to-metal bond lines this allowance is 0.0009 + 0.0001, -0.000 inch, and for metal-to-core bond lines the allowance is 0.0015 + 0.005 inch.

D. Inspection - After machining, the edges of the cell walls at the core face cannot be split, bent over, or otherwise deformed. The machined face must be free of burrs, particles, and partially severed foil.

5.1.6.2 Trimming of honeycomb core

- (a) Do not apply sufficient force to cause crushing or buckling (Columnar failure) in any area except that to be completely removed by subsequent trimming.

- (b) Core details that have become delaminated during machining are to be considered for rejection or repair.
- (c) Cell wall extending more than 0.010 inch below the plane of the honeycomb part face or more than one cell in width or more than 1 inch in length, except on areas that are to be subsequently reinforced by a foamed adhesive or removed by trimming, are subject to rejection or approved deviation in accordance with the provision of this document.
- (d) Cut the core in a manner so that the ribbon direction in detail parts does not vary more than ten degrees from that specified on the applicable drawing. When not specified on the drawing, ribbon direction is optional.
- (e) The core may contain only those splices specifically allowed by the applicable engineering drawing.

5.2 Cleaning - Honeycomb core is clean when received and with proper handling, will require only vapor degreasing in accordance with Specification MS 150.0 except as indicated in "Cleaning for Adhesive Bonding," M-ME-MPROC 005.5B-3.

5.2.1 Handling of cleaned parts - After cleanliness inspection, all parts must either be assembled or primed with adhesive within 8 hours after drying. If the time limit of 8 hours is exceeded, the parts must be recleaned. Parts cleaned or surface treated as above cannot be touched by bare hand in any subsequent operation up to and including assembly of parts. Wear clean white gloves at all times while handling parts. These gloves must be discarded at the first indication of contamination. At the time of assembly, the entire part must be clean and free of dust, oily material, wax, cleaning agents, etc. It is essential that bond areas are not contaminated in any manner. Any part showing evidence of contamination of the bond area will be rejected, recleaned, and retreated as specified herein.

NOTE: Cleaned details except core details, including process control test assembly details which, because of impending work stoppage, cannot be processed, assembled, and cured prior to work stoppage, must receive a spray coat of adhesive primer within 8 hours.

5.3 Preparation and application of adhesives

5.3.1 Mixing - Agitate each full container of liquid adhesive, after it is brought to room temperature, for at least 5 minutes before it is used. Any adhesive containing lumps or showing an indication of lumps or areas of solidification after agitation is to be rejected.

5.3.2 Adhesive primers - When adhesive primers are used, only those adhesive primers that are compatible with the adhesive and are specified may be used.

5.3.3 Spray application of liquid adhesive or adhesive primers - Unless otherwise specified, liquid adhesive primers will not be applied to honeycomb core. The procedures for application are as follows:

- (a) Apply a smooth film of thinned adhesive primer to each faying surface of all parts being bonded. The adhesive primer film must extend at least 1/8 inch beyond the bond area where possible. Small areas may be brush coated if uniform thickness is maintained.
- (b) Apply the adhesive primer in one coat consisting of two approximately equal cross passes. The coating must be applied "wet" to obtain a continuous film. Thickness of the dried film should be as specified for the particular adhesive used.
- (c) Air dry adhesive primer coat, if specified, in accordance with the manufacturer's data sheet or as defined by S&E-ME-MM.
- (d) Following the air dry, if specified, precure the primer in a dust-free oven at the temperature recommended by the adhesive manufacturer or as specified by S&E-ME-MM.
- (e) After precuring, the primed part should not be exposed to the atmosphere for more than 24 hours before assembly of parts. Parts may be stored up to 90 days after precuring at room temperature (90° F maximum) provided the part is carefully wrapped in wax-free kraft paper. Ink stamp the date and time of adhesive primer application on the part outside of the bond area and on the outside of the wrapping. In event the 90 day storage time limit is exceeded, the part may not be processed except as specifically authorized by S&E-ME-MM.
- (f) Remove any overspray of liquid adhesive on exterior skin surfaces with MEK or similar approved solvent before the coating has completely dried. Adhesive in the bond areas must not be diluted by solvent used to remove overspray.

5.3.4 Application of film adhesives

5.3.4.1 Film adhesive with protective liners on both sides should be applied as follows:

- (a) Remove the parting liner from one surface, after the adhesive is brought to room temperature, where applicable.
- (b) Apply the film adhesive on the selected primed area of the interfaces of each bond joint.
- (c) Remove the remaining protective liner just prior to assembly of parts.

5.3.4.2 Film adhesive with one side covered by protective liner should be applied as follows:

- (a) Cut the amount of film adhesive needed from the roll leaving the protective liner in place.
- (b) Apply the film to one side of the faying surface by rolling the tacky surface against the metal.
- (c) Remove the protective liner just prior to assembly of parts.

5.3.4.3 Detailed procedure for application of film adhesive

- (a) Roll the film adhesive into place so as to exclude air bubbles or other areas of entrapped air against skin material. The adhesive may be tacked to metal parts by touching several spots with a clean, Teflon covered, hot tool such as a soldering iron or hot air gun. The temperature of the tacking tool shall not exceed 200°F.
- (b) Trim the adhesive, leaving approximately 1/8 inch beyond the perimeter of the joint surface when permitted by the layup.

NOTE: If more than one piece of film is used on a part, butt splices should be used and gaps should not exceed 1/32 inch. Whenever possible, film splices should not be located closer than 1 inch to material splices.

- (c) Parts should be bonded within the specified layup time as specified in the appropriate adhesive data sheet. If this exposure time is exceeded the adhesive film should be removed and replaced with new film.

- (d) Adhesive film that has been exposed to room temperature for a cumulative total exceeding the maximum time allowed by the adhesive data should be discarded.
- (e) Film adhesive should not be touched by bare hands. Clean, white gloves should be worn at all times while applying adhesive, peeling off protective liner, and assembling of parts.
- (f) Cutting tools, templates, and tools used for laying out, cutting, or handling of film adhesives should be vapor degreased or solvent cleaned as frequently as necessary to insure freedom from contamination.

5.4 Curing of adhesives

5.4.1 General

5.4.1.1 A cure cycle is defined as the interval elapsing between the time that the adhesive bond is heated to 150°F and the time that it is cooled to 150°F after the cure period.

5.4.1.2 A cure period is defined as the time at the cure temperature.

5.4.1.3 The cure cycle for each adhesive should be completed within the time limits recommended by the adhesive manufacturer or as indicated by S&E-ME-MM.

5.4.1.4 The cured adhesive bond should not be subjected to stress while removing the assembly from the curing facility or tool unless the temperature of the bonds are below 150°F and then only if necessary to free the assembly.

5.4.1.5 Parting agents should be used where required on all tools.

5.4.2 Autoclave bonding

5.4.2.1 General - The curing of parts by means of flexible bags or blankets permits uniform application of pressure and the autoclave provides uniform temperatures. Such bags or blankets may be made of fiberglass cloth, osnaburg cloth, or rubber sheeting. "Bridging" should be avoided as it prevents proper application of pressure and may result in a faulty bond. "Free floating" pressure bars or rings may be used to better control pressure direction.

NOTE: Extreme caution should be exercised in autoclave bonding since serious and sometimes unexplainable fires may be experienced. The combination of compressed hot air and combustible materials (such as rubber or plastic bags or blankets, oil vapors from the compressor, and solvent vapors from the adhesive that may be squeezed through a defective bag or blanket) is exceedingly dangerous and may be a potential explosion hazard. The maintenance of a vacuum pump on the vent from the bonding fixture would lessen the latter hazard considerably. The use of an inert gas pressurizing medium would also be advisable to prevent fires. Nitrogen is used in all autoclaves at MSFC.

5.4.2.2 Bags or blankets - The purpose of the bag or blanket is to provide a flexible impermeable barrier between the pressure medium and the parts being bonded. The flexible bag or blanket also provides a uniform pressure with direction normal to the adhesive plane.

5.4.2.3 Assembly

- (a) Place detail parts in the bonding fixture using pins in rivet holes or other suitable means for alignment.
- (b) Install thermocouples in the assembly as specified.

5.4.2.4 Bonding procedure - When an autoclave is to be used in the curing of adhesives, the following procedure should be followed:

NOTE: It is understood that the following procedure is not all inclusive, and will not apply to all configurations. If sufficient information is not available, call S&E-ME-MM.

- (a) Place a bleeder material, such as fiberglass cloth or osnaburg cloth, over the assembly and over edges and corners as required.
- (b) Cover the sealed assembly with a blanket that is sealed to the fixture with sealant paste, or equivalent.
- (c) Check the sealed assembly for leaks by attaching a vacuum line and applying a maximum of 14 inches of mercury. Close the vacuum line and check the indicating gage attached to the vacuum blanket. A vacuum drop of 2 inches or more per minute will indicate excessive leakage. Any leaks located should be sealed and rechecked.

- (d) Place the sealed assembly in the autoclave, join the vacuum couplings, and determine the effectiveness of the seal.
- (e) The thermocouple is connected to the indicator.
- (f) Check thermocouple junction with indicator leads for proper operation.
- (g) Close and seal autoclave. Apply autoclave pressure before the temperature reaches 150°F. Release vacuum and vent assembly interior to atmosphere when the pressure in the autoclave reaches 10 to 15 pounds per square inch (psi) and before the temperature exceeds 150°F.

NOTE: Vacuum may be used during the complete cycle when a nonfoaming type adhesive is being used.

- (h) Raise the temperature to the predetermined curing level at a suitable rate for the particular adhesive used. The autoclave may be preheated prior to loading providing the pressure is applied before the bond line reaches 150°F.
- (i) Maintain temperature for the time specified on the applicable data sheet or as specified by S&E-ME-MM.

5.4.2.5 The assembly should be cooled to 150°F or less while under bonding pressure prior to removal from bonding fixture and should be accomplished in a manner so as to assure uniform cooling of entire assembly to avoid warpage.

5.4.3 Press bonding - The source of pressure is normally a hydraulic piston and stops should be used in cases where the shape of the parts may be distorted. In using stops, metal tolerances and bonding pressures should be carefully considered to insure the integrity of the completed part. To avoid nonuniform pressure on flat parts, it is permissible to use liquid-filled metal bladders between the platen and the surface being treated. Bonding in platen presses or other unit tool will normally be limited to first stage bonding.

5.4.4 Curing time and temperature - The selection of the curing time and temperature and even the rate of heating required are dependent on the adhesive formulation, the type of joint, and the service condition expected for the bond. The adhesive manufacturers recommendation should be carefully considered in establishing the curing conditions to be used in fabrication of the bonded assemblies. The elevated-temperature-setting adhesives are usually cured at 250 to 350°F for 30 minutes to 2 hours. Certain formulations of the epoxy-resin adhesives can be cured at 180 to 200°F. Moderately strong bonds are obtainable

with some special formulations of this type adhesive when cured at normal shop temperatures. For a particular application, the curing time and temperature must be obtained from the applicable manufacturer's adhesive data sheets or from S&E-ME-MM. These adhesive data sheets are subject to revision.

5.4.4.1 Unless otherwise specified, at least one thermocouple in the upper skin bond and one in the lower skin bond should be used for each 10 square feet, or fraction thereof, of the plan form area of an assembly or for each 10 lineal feet, or fraction thereof, of peripheral length of the assembly, whichever is greater.

5.4.4.2 The thermocouples used should be fabricated from fine iron-constantan wire (No. 30 maximum) and should be embedded in the adhesive bond or in the adhesive flash immediately adjacent to the edge of the metal from under which the adhesive extends. Each thermocouple used should be accurate to within $\pm 5^\circ$ at 335°F .

5.4.4.3 Each thermocouple circuit should be checked for continuity immediately prior to starting a bonding cycle.

5.4.4.4 The rate of heating from 150°F to the minimum cure temperature should be as uniform as possible over the entire skin area of both sides of each assembly and should not exceed 10°F per minute. Difference in temperature at measuring points should not exceed 20°F at any time.

5.4.4.5 The curing temperature of each adhesive bondline thermocouple installation should be continuously monitored and permanently recorded, by an accurately calibrated temperature recorder, during the entire cure cycle.

5.4.4.6 At the completion of its cure period, each bonded assembly should be air cooled as rapidly and at as uniform a rate as practicable.

5.4.5 Bonding pressure - Adequate pressure should be maintained during the final cure of the adhesive bond to (1) obtain uniform adhesive bond thicknesses, (2) overcome internal pressure exerted by release of adhesive solvents, (3) overcome viscosity of adhesive film at the curing temperature, and (4) overcome the surface imperfections (within limits) between mating surfaces or the lack of flatness in the skin materials. The pressure required for bonding is a constant within limits for any one adhesive, but may vary for different assemblies. Therefore bonding pressure should be established for each project as the projects is designed and scheduled. Production parts shall be cured to ± 5 psi of that established pressure.

5.4.5.1 Bonding pressure control - Constant pressure should be maintained on the entire bond area during the cure cycle. Pressure should be applied in such a manner so as to prevent lateral movement of the assembly parts or displacement of the adhesive. Curing pressures should be applied before the adhesive reaches a temperature of 150°F. Adhesive bonds, other than core splices and core-to-extrusion bonds, should be cured under fluid pressure applied to the assembly and tools through a flexible blanket. The flexible blanket should be in direct contact with one surface of the assembly being bonded or should apply the pressure through any material used between the part and the blanket, that will uniformly transmit the fluid pressure. Wrinkles should be kept to a minimum. Bridging over step configurations should be avoided.

5.4.6 Post cure bonding - When room-temperature-cure edge filler or potting compound must be employed, a suitable two-part thixotropic paste should be used. Since in most cases this is a catalyzed material, it must be applied within 1 hour or less after mixing. Only sufficient material should be mixed for the specific job. Storage under refrigeration after mixing will extend the working or pot life.

5.4.6.1 Filling cavities - For filling of cavities, injection by means of a Pyles gun or equal should be employed. Where potting for inserts, etc., is indicated, sufficient compound should be used to completely encapsulate the insert and fill the hole or cavity. Any excess compound must be removed immediately.

5.4.6.2 Fastener attachment - When attaching details with blind fasteners, all holes drilled in the honeycomb core to provide clearance for the blind fasteners should be reinforced by injecting the potting compound under 5 psi pressure to fill core cells damaged by drilling. Fasteners must be installed within 60 minutes after injection. Clearance holes are to be drilled a maximum of 1/4 inch in excess of the extended length of the fastener into the core.

5.4.7 Room-temperature-cure edge filler or potting compound - When room-temperature-cure edge filler or potting compound is to be used, curing temperatures, cure time, and cure pressures are to be in accordance with the applicable manufacturer's adhesive data sheet or as specified by S&E-ME-MM.

6. FINISH REQUIREMENTS

6.1 Finish and corrosion protection

6.1.1 Leave the cement flash to seal the joints of the completed assembly whenever possible. If the flash must be removed, trim in a manner which will not cause delamination of the bond.

6.1.2 Aluminum alloy assemblies require no bond protection other than that offered by the cement flash, except that edges trimmed in a manner which exposes bare metal or destroys cement flash must be protected.

6.1.3 Complete the assembly in accordance with the applicable finish specification.

6.2 Packaging and storage

6.2.1 After the completed assembly has been accepted by inspection, give it a heavy coat of Amercoat No. 1133 blue or equivalent strippable coating if specified.

6.2.2 After protective coating has been applied, package the assembly in a suitable container to prevent damage during storage, transportation and/or shipment.

6.3 Tooling

6.3.1 Core splicing bonding - Provide a fixture or jig to hold core sections in firm contact in all bond areas and in proper alignment with each other during splice cures. Core splices may be bonded with core in a vertical or horizontal position.

6.3.2 Final assembly bonding - Provide a final assembly bonding tool with means for locating and indexing core and other detail parts or subassemblies.

6.3.3 All tooling areas that contact adhesive must be coated with approved parting agents, such as Teflon spray or cellophane, which will not contaminate the bond.

6.4 Bonding Facilities requirements

6.4.1 All operations, after cleaning of parts through sealing of the assembly and vacuum check is to be conducted in an area where cleanliness is carefully maintained and controlled by policing the area and filtering of all incoming air to 100 microns absolute. Temperature of the area must be controlled to stay within the range of 65 to 75°F and the relative humidity maintained at less than 70.

6.4.2 All personnel must wear clean, white gloves while handling parts. Only authorized personnel are permitted in the clean area. Silicone greases or lubricants, talc and similar materials are not to be used in the clean room for any purpose.

6.4.3 Make a record and keep a permanent file, listing material used, exact steps in metal surface preparation, pertinent assembly data, serial numbers of bonding fixture used in bonding each component, the pressure, temperature, and time at pressure and temperature of each component during curing cycle. The record pressure temperature, and time at pressure and temperature should be made on automatic recording devices. Temperature devices should be inspected every 30 days. All others at 6-month intervals.

6.5 Workmanship - Honeycomb sandwich components are to be fabricated in accordance with the best known commercial practice for obtaining the highest quality product available from the materials used. On all aero-dynamic and structural surfaces, components must be smooth, free from scratches or dents, and otherwise completely suitable for space launch vehicle applications.

7. MANUFACTURING CONTROL

7.1 Preprocess test specimens

7.1.1 Preparation of test specimens - Prior to production of hardware, fabricate six sandwich panels of 3 inches in width by 16 inches in length to be used as test specimens. These specimens should be fabricated using the same alloy, facing thickness, core thickness, core cell size, density, adhesives, cleaning and curing procedures and conditions as required for the production item. The facings are to be 16 1/2 inches, unless specified on drawing. Bond the facings to the core with 1/2 inch facing sheet extending alternately from each end to accommodate a drum peel tester. The ribbon of the core is to be parallel to the 16 inch length of the specimen. (As specified by the drawing or by S&E-ME-TP).

7.1.2 Sandwich flexure test - All six specimens fabricated in accordance with paragraph 7.1.1 are loaded to failure at a constant rate of the movable head of the testing machine of not less than 0.015 nor more than 0.020 inch per minute. Load, using the fixture shown in Figure 9-2. Tests are conducted at room temperature (70 to 80°F) unless otherwise specified in design drawings. The load at failure should be equal to or greater than the minimum load requirement determined from the chart of Figure 9-1. Records are to be maintained of the load at failure and of the type of failure. Where two different facing thicknesses are used in the specimen, the thinner facing is placed upward in the beam test fixture.

1. CORE SHEAR BASED ON MINIMUM STRENGTH REQUIREMENT VALUES. FOR ALUMINUM CORE AND SPAN PARALLEL TO L DIRECTION.

2. LOAD CALCULATED BY FORMULA $P = 2 \times b \times S_s \times h$, WHERE:

P = LOAD IN POUNDS; b = PANEL WIDTH IN INCHES;

S_s = CORE SHEAR IN POUNDS PER SQUARE INCH; h = PANEL THICKNESS IN INCHES.

3. TENSILE OR COMPRESSIVE STRESS IN FACINGS CALCULATED BY FORMULA $C_s = PA/2bh$, WHERE C_s = COMPRESSIVE OR TENSILE

STRESS IN POUNDS PER SQUARE INCH; P = LOAD IN POUNDS;

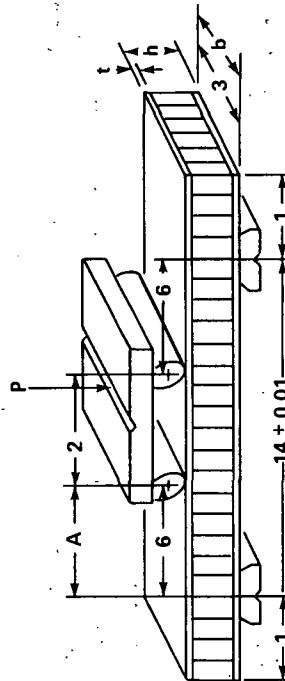
A = OFFSET DISTANCE IN INCHES; b = PANEL WIDTH IN

INCHES; t = FACING THICKNESS IN INCHES; AND h = PANEL THICKNESS IN INCHES.

4. CHART BASED ON TESTING OF A 16 INCH LONG (L DIRECTION) BY 3 INCH WIDTH SPECIMEN, AND AN OFFSET DISTANCE OF 6 INCHES FOR ALL TESTS.

5. EXAMPLE: IF $h = 1.5$ INCHES, $t = 0.032$ INCH AND DENSITY OF HONEYCOMB CORE (ρ) IS 4.5 POUNDS PER CUBIC FOOT, CORE IS 5052-H39 AL AND THE FACINGS ARE 7075-T6 AL, DETERMINE MINIMUM ACCEPTABLE LOAD (P) AT FAILURE OF SPECIMEN.

SOLUTION: ENTERING CHART AT $\rho = 4.5$ PROJECT LINE TO CURVE FOR 5052-H39 ALUMINUM, THEN VERTICALLY TO $h = 1.5$ THEN HORIZONTALLY TO $P = 2150$ POUNDS. THIS IS THE MINIMUM ACCEPTABLE LOAD FOR THE SPECIMEN AT FAILURE OF BOND. TO FIND THE COMPRESSIVE STRESS IN THE UPPER FACING AT FAILURE OF THE BOND CONTINUE THE PROJECTION LINE FROM P , HORIZONTALLY TO $h = 1.5$, THEN VERTICALLY TO $t = 0.032$, THEN HORIZONTALLY TO $C_s = 44\ 500$. SINCE THIS COMPRESSIVE STRESS IS CONSIDERABLY LESS THAN THE YIELD STRENGTH OF 7075-T6 ALUMINUM, THE FACINGS SHOULD NOT WRINKLE OR BUCKLE UNDER A LOAD OF 2140 POUNDS.



t - FACING THICKNESS

h - SPECIMEN THICKNESS

P - LOAD IN POUND

END SUPPORT PLATES ARE 1 BY 3/4 INCHES WITH GROOVES FOR ALIGNMENT. LOADED EDGES ARE ROUNDED TO 0.06 INCH RADIUS.

LOAD BARS ARE 1/2 INCH ROUND.

DIMENSIONS IN INCHES.

TOLERANCES: AS INDICATED

Figure 9-2. Beam flexure test aluminum facings and core sandwich structures.

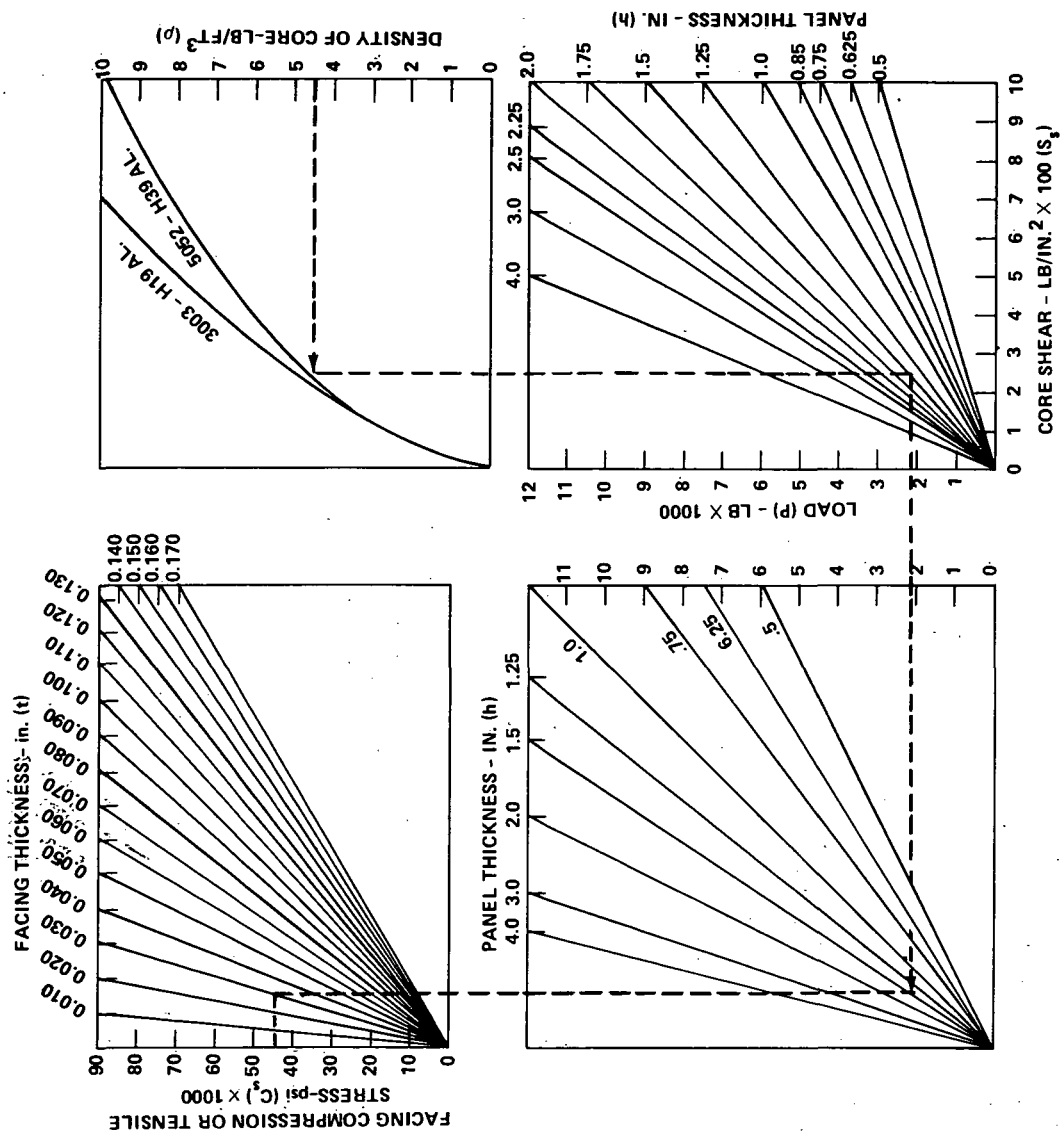


Figure 9-2. (Concluded)

7.1.3 Peel test - A peel test should be conducted on one of the failed beam specimens at room temperature, using a climbing-drum peel tester as illustrated in Figure 9-2. The test will be made by clamping the sandwich specimen securely in place and loading the apparatus in tension at a head speed of 1 inch \pm 0.001 inch per minute. A head speed of 1 inch per minute will peel the sandwich facing at a rate of 4 inches per minute. The room temperature requirements are specified on the manufacturer's data sheets or may be specified by S&E-ME-MM.

7.2 In-process test specimens

7.2.1 Source - Production bonded assemblies shall incorporate, where possible, extensions or coupons which can be cut from the assemblies after bonding which will be representative of the bonds in the assembly and of the test specimens of 7.1. Where extensions cannot be incorporated in the parts, standard specimens (3 inch x 16 inch) shall be fabricated to simulate such extensions; however, these extensions must be bonded at the same time using same adhesive material, in the same equipment and under the same conditions as for the assembly they represent. These in-process test specimens shall be tested for flexural strength and peel strength in the exact manner and conditions as the pre-process specimens of paragraph 7.1.

7.2.2 Production stoppage - When a production part has been rejected because of failure to meet the requirements of paragraph 7.2, work on the production item shall be stopped and not resumed until the cause for the improper processing has been determined and corrected.

8. QUALITY ASSURANCE PROVISIONS

8.1 Tests - The tests used to determine compliance with the requirements of this process are of two types; acceptance tests and process control tests.

8.2 Materials acceptance tests and test methods

8.2.1 Metal-to-metal adhesives

8.2.1.1 Room temperature tensile shear strength - Tensile shear strength is to be determined in the manner specified in Specification MMMA-132.

8.2.2 Core-to-skin adhesives

8.2.2.1 Room temperature sandwich peel strength - Room temperature sandwich peel strength is to be determined in the manner specified in paragraph 4.6.1 of Specification MIL-A-25463 (ASG).

8.2.3 Honeycomb core

8.2.3.1 Core density and specific gravity - Core density and specific gravity are to be determined in the manner specified in MIL-C-8073D.

8.2.3.2 Core compression - Determine core compressive properties in the manner specified in paragraph 5.1.4 of Standard MIL-C-7438D.

8.2.3.3 Core shear - Determine shear properties in the manner specified in paragraph 3.1.3 of Standard MIL-C-7438D.

8.2.3.4 Core tensile strength - Determine core tensile properties in the manner specified in paragraph 5.1.6 of Standard MIL-C-7438D.

8.3 Process Control Tests

8.3.1 Nondestructive tests

8.3.1.1 Visually examine all sandwich construction immediately after manufacture to determine that it meets all requirements of the applicable engineering drawing with respect to configuration, dimensions, uniformity, and appearance. Bonded panels or components which show evidence of blisters in the facings upon removal from curing press or bag will be rejected or will have such areas marked for ease in future location for repair.

8.3.1.2 Sonic tapping - Check all sandwich construction for void in the adhesive bond between the facings and core by means of ultra-sonic devices.

9. NOTES

9.1 Intended use - This manufacturing process has been coordinated within the Process Engineering Laboratory of the George C. Marshall Space Flight Center and is intended for use in process control in the fabrication of bonded aluminum honeycomb sandwich structures for use in space launch vehicle applications.

9.2 Cutting of test panels and specimens - Extreme care must be exercised in the cutting of test specimens from panels to minimize heating and

vibration. If this is not done, damage to bonded joints will occur and will result in low or erroneous test results.

9.3 Deviations - Any deviation from this manufacturing process will require written concurrence from the Process Engineering Laboratory. An emergency concurrence may be obtained by telephone, but it must be confirmed in writing.

STRUCTURAL ADHESIVE BONDING METAL TO METAL STRUCTURES

1. SCOPE

1.1 Scope - This manufacturing process specifies the Process Engineering Laboratory requirements for the fabrication of metal to metal bonded components using structural adhesives. Cleaning requirements for the various materials being bonded are outlined in the applicable cleaning specifications for the type of metals being bonded.

1.2 Applicability - The provisions of this specification are applicable to all metal to metal composite structures fabricated for or by the Process - Engineering Laboratory. The process for the bonding of honeycomb structures is contained in M-ME-MPROC-005.5B-1.

1.3 Restrictions - No other materials or processes may be used as alternates or equivalents to those required by this document without prior written approval of the S&E-ME-M Division.

2. APPLICABLE DOCUMENTS

2.1 Governmental - The following documents form a part of this specification to the extent specified herein.

Specifications

TT-M-261	Methyl-Ethyl-Ketone, (For use in Organic Coatings).
CCC-C-440	Cloth, Cheesecloth, Cotton, Bleached and Unbleached.
UU-P-268	Paper, Kraft, Untreated, Wrapping.
MIL-G3866	Gloves, Cloth, Cotton, Knitted, Lightweight.
MMM-A-132	Adhesives, Heat Resistant, Airframe Structural, Metal to Metal.

Federal Standards

- FED-STD-175 Adhesives, Methods of Testing
- FED-STD-264A Clean Room Requirements (Class 100 000).

Publications

National Aeronautics and Space Administration

- NPC 200-3 Inspection System Provisions for Suppliers of
Space Materials, Parts, Components, and
Services.

3. EQUIPMENT

- 3.1 Autoclave or press - capable of providing 100 psi and 350° F controlled heat cycle.
- 3.2 Primer Drying Oven
- 3.3 Spray Gun - Devilbiss gun, fluid needle MBC 444 FX, fluid tip AV-15-FX, and air cap number 36, or equal.
- 3.4 Pressure Bags - Materials used must provide a flexible impermeable barrier between the pressure medium and the part being bonded. A suitable bleeder material should be used between the part and the pressure bag.
- 3.5 Vacuum Pump - A vacuum pump of suitable capacity is required for evacuating the sealed assembly.
- 3.6 Cleaning Tanks, Rinse Tanks, and Etching Tanks - Cleaning, etching, and rinse tanks are required for surface treat and cleaning. Ref: Paragraph 5.2.
- 3.7 Fixtures - Bonding and/or lay-up fixtures should be provided as required.

4. MATERIALS

4.1 Adhesives - The adhesives used in the production of bonded metal to metal composite structures must meet all the requirements of the detail drawings or as specified by the Project Engineer.

4.1.1 Certification of Adhesives - The vendor is required to supply documents containing the following information:

- Vendors adhesive batch* number.

* A batch is defined as that quantity of material which has been manufactured at one time or subjected to some unit chemical or physical mixing process intended to make the final product homogenous.

- Date of manufacture.
- Date shipped from vendor.
- Adhesive Storage requirements

4.1.2 Control Record - Upon receipt of a new batch of adhesive from the manufacturer, a record of that batch must be originated and maintained until the batch is entirely used up or discarded. This record should contain the following information:

- Manufacturer's batch number.
- Date of manufacture.
- Expiration date of manufacturer's guaranteed shelf life.
- Date of acceptance testing.
- All acceptance test values.
- Storage temperature range (minimum, maximum).
- Scheduled reinspection date (during storage).
- Date and time into and out of refrigerator. (If applicable)**
- Dates of evaluations for release for production use.

- All production evaluation test values.
- Dates of issue for production.

** Single component and film adhesives.

4.1.3 Acceptance Testing - Within 72 hours after the receipt of each new batch of adhesive, the roll or container is to be exposed to room temperature ($70 \pm 5^\circ \text{F}$) until moisture condensation ceases. Remove a sample from this roll or container and use to bond test specimens. Seal the adhesive material and return to storage as soon as possible. These test specimens must be subjected to the tests outlined in the data sheets for the particular adhesive, or per the instructions of S&E-ME-MM.

4.1.4 Periodic Recertification - Periodic recertification is required on all adhesives that have not been released for production use within the guaranteed shelf life. Periodic recertification consists of subjecting samples to the tests specified in the data sheets, or as specified by S&E-ME-MM.

4.1.4.1 Room Temperature Tensile Shear Test - For production parts, a minimum of two tensile shear specimens per platen load are required. These specimens go to the production evaluation group for testing. The specimens must meet the strength requirements of the applicable specifications or that imposed by S&E-ME-MM.

4.1.5 Preproduction Evaluation - Adhesives used in the production bonding of metal to metal composite structures must be tested as specified herein within 168 hours preceding use. No acceptable lot of adhesive is to be used in production of parts later than 168 hours after it was sampled for such testing, unless it is again sampled, tested, and approved for use.

4.1.5.1 Metal to Metal "T" Peel Test - Prepare one bonded metal to metal test panel as shown in Figure 9-3. Cut 1/2 inch from each side and discard. Cut six 1-inch wide "T" peel test specimens from each panel in accordance with Figure 9-3. Five specimens are tested at ambient temperature. The samples should be peeled at least 3 inches at a rate of 8 to 14 inches per minute. Record the peel value continuously, or take 8 to 10 readings during peeling at 1/4 inch intervals and average to obtain the peel value.

4.1.5.2 Mismatches - For complex assemblies, vinyl impression checks should be made to adequately determine mismatches between faying surfaces. All prefitted parts must be completely identified with the assembly of which

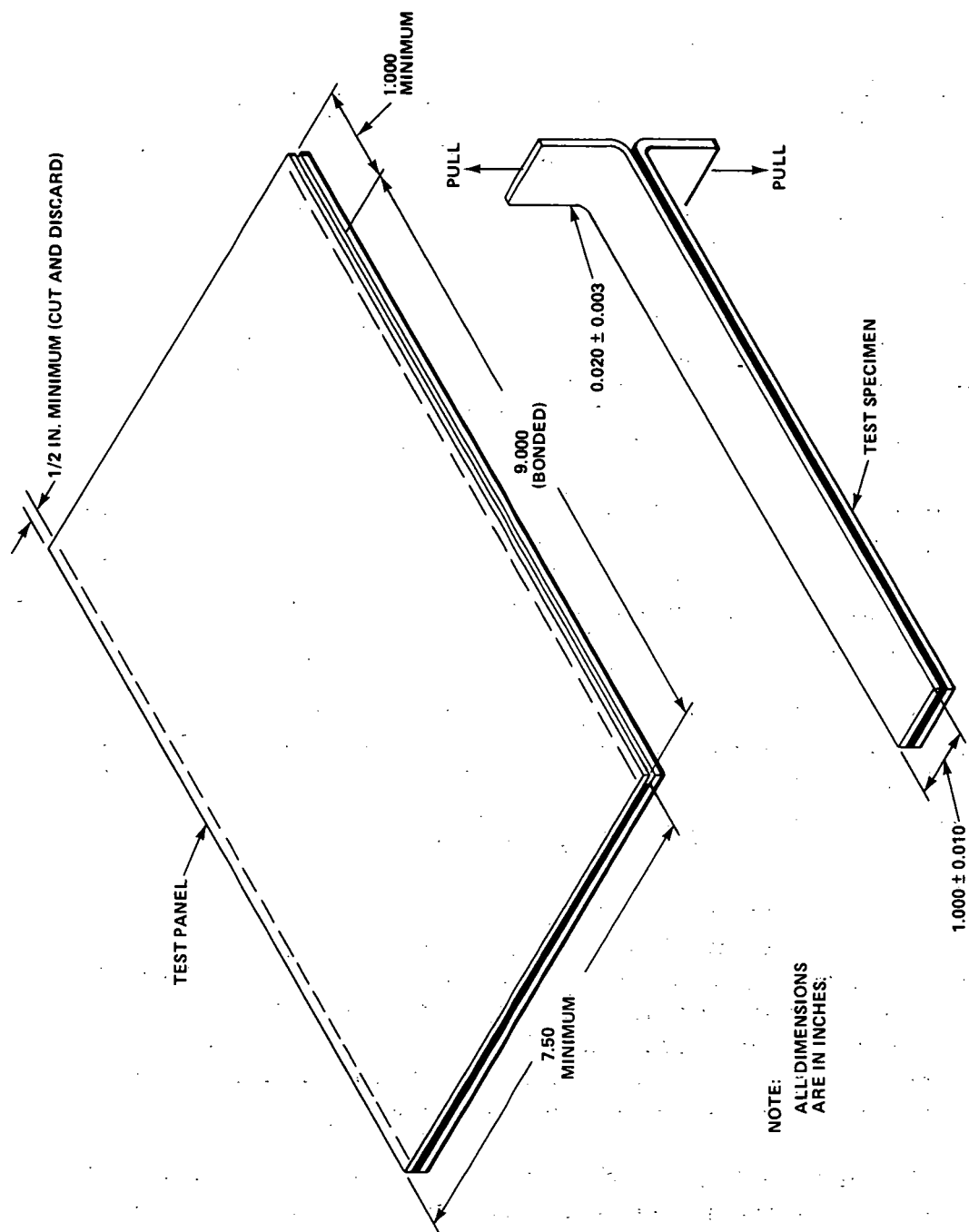


Figure 9-3. "T" - Peel test specimen.

they are a part." Mismatches in excess of 1/2 the nominal thickness of the adhesive film will require additional layers of adhesive. The number of additional layers permissible are limited, and will be specified on the drawing, or by S&E-ME-MM.

4. 1. 6 Venting - One of the two faying surfaces is to have an adequate number of perforations or vent holes when the overlap of bonded surfaces exceeds 1 inch and when adhesives are used that liberate gases in the curing process.

4. 2 Sheet and Plate - Face sheets and formed parts to be bonded must be fabricated from materials that meet the requirements of the applicable material specification.

4. 2. 1 Storage - Face sheet material must be kept stored, in a protected area.

4. 2. 2 Handling - Sheet material and formed parts require protection and must be handled in such a manner as to prevent scratches, dents, abrasions, warping, or bowing.

4. 2. 3 Protection - Sheet material surfaces are to be protected at all times to prevent damage and contamination from any source.

4. 2. 4 Acceptance Testing - In addition to certification by the vendor, the following tests may be required, at the discretion of S&E-ME-MM.

- Chemical analysis.
- Mechanical properties (tensile strength, yield strength, elongation and hardness).
- Resistivity.
- Metallographic determination of homogeneity.

4. 3 Miscellaneous Materials

4. 3. 1 Methyl-Ethyl-Ketone - Methyl-Ethyl-Ketone (MEK) used in the performance of the processes specified herein must conform to the requirements of Specification TT-M-261.

4.3.2 Wrapping Paper - Wrapping paper used in the performance of the processes specified herein must conform to the requirements of Specification UU-P-268.

4.3.3 Gloves - Cotton, or plastic gloves used in the performance of the processes specified herein must conform to the requirements of Specification MIL-G-3866, or an equivalent.

4.3.4 Cleaning Cloths - Cleaning cloths used in the performance of the processes specified herein must conform to the requirements of Specification CC-C-440.

5. BONDING PROCESS

5.1 Precleaning Requirements

5.1.1 Contact Surfaces - Contact surfaces of all parts should be relatively free from burrs, dimples, scratches, steel stamped part numbers, or other surface defects.

5.1.2 Edge Chamfer - The edge chamfer or radius resulting from the removal of burrs cannot exceed 0.010 inch.

5.1.3 Forming Dies - Forming dies used to form parts for adhesive bonding are to be lubricated with a compatible material containing no silicones, e.g., Everlube T-50 Formkote.

5.1.4 Identification - Identify all parts with the assembly of which they are a part.

5.1.5 Prefitting - All surfaces to be joined by adhesive bonding must be prefitted so that only a minimum pressure is required.

5.2 Cleaning - All details are to be cleaned in accordance with M-ME-MPROC 005.5B-3 and -4 cleaning specifications available through S&E-ME-MM.

5.2.1 Handling of Cleaned Parts - After cleanliness inspection, all aluminum parts must either be assembled or primed with adhesive within 8 hours after drying. If the time limit of 8 hours is exceeded, the parts must be recleaned. Other metals must be primed immediately. Parts

cleaned or treated as above cannot be touched by bare hands in any subsequent operation up to and including assembly of parts.

Clean, white gloves are to be worn at all times while handling parts. Discard these gloves at the first indication of contamination. At the time of assembly, the entire part must be clean and free of oily material, wax, cleaning agents, etc. Any part showing evidence of contamination of the bond area will be rejected, recleaned, and re-treated as specified herein.

5. 3 Preparation and Application of Adhesive - The compound and activator shall be mixed in containers of a nonpourous material such as polypropylene, polyethylene, glass, or metal.

5. 3. 1 Mixing - Each full container of liquid adhesive, after it is brought to room temperature, must be mixed adequately before it is used. Any adhesive containing lumps or showing any indication of lumps or areas of solidification after agitation will be rejected. See manufacturer's specification for open assembly time, storage and pot life.

5. 3. 2 Adhesive primers - Where adhesive primers are used, only those adhesive primers that are compatible with the adhesive and are specified are to be used.

5. 3. 3 Spray application of adhesive or adhesive primer

(a) Using a suitable line pressure, adjust spray gun to deposit a wet film. Cobwebbing and uneven coating shall be minimized. Air used for spraying purposes shall be filtered through oil traps to reduce concentration. The adhesive or adhesive primer must be applied in a clean spray booth designated and used for this purpose only. When the spraying operation is completed, clean the spray equipment with MEK or similar approved solvent.

(b) The adhesive or adhesive primer must be relatively free of all foreign material, moisture, deposits from adjacent spray operations, etc. The coating cannot be touched with bare hands. After spraying, parts must be protected until assembly. Contamination of the faying surfaces cannot be tolerated. Application of excessive adhesive, adhesive primer, or the formation of large droplets on the surface, will be cause for rejection and reprocessing of the affected part. Adhesive primer must be removed from such parts by suitable means prior to recleaning or retreating.

5. 3. 3. 1 Application techniques

(a) Apply a smooth film of thinned adhesive primer to each faying surface of all parts being bonded. The adhesive primer film must extend at least 1/8 inch beyond the bond area where possible. Small areas may be brush coated if uniform thickness is maintained.

(b) Apply the adhesive primer in one coat consisting of two approximately equal cross passes. The coating has to be applied "wet" to obtain a continuous film. Thickness of the dried film must be as specified for the particular adhesive used.

(c) Following the air dry, if specified, the primer must be precured in an oven at the temperature recommended by the adhesive manufacturer, or procuring activity.

(d) After precuring, the primed part cannot be exposed to the atmosphere for more than 24 hours before assembly of parts. Parts may be stored up to 90 days, or as specified by manufacturer, after precuring at room temperature (90° F), provided the part is carefully wrapped in wax-free paper or equivalent, per UUP-268. The date and time of adhesive primer application must be ink-stamped on the part outside of the bond area or on the outside of the wrapping. In event the 90-day storage time limit is exceeded, the part may not be processed except as specifically authorized by S&E- ME-MM.

(e) Remove any overspray of liquid adhesive on exterior skin surfaces with MEK or similar approved solvent before the coating has completely dried. Adhesive in the bond areas must not be diluted by solvent used to remove overspray.

5. 3. 4 Application of film adhesives

5. 3. 4. 1 Film adhesive with protective liners on both sides - Apply film adhesives with protective liners on both sides as follows.

(a) Remove the parting liner from one surface, after the adhesive is brought to room temperature, where applicable.

(b) Apply the film adhesive to the primed area of the interfaces of each bond joint.

(c) Remove the remaining protective liner just prior to assembly of parts. Dry ice may be used as an aid in removal of protective liner.

5.3.4.2 Roll film adhesive with one side covered by protective liner - Roll film adhesive with one side covered by protective liner is to be applied as follows:

(a) Cut the amount of film adhesive needed from the roll leaving the protective liner in place.

(b) Apply the film to one side of the faying surfaces by rolling the tacky surface against the metal.

(c) Remove the protective liner just prior to assembly of parts. Dry ice may be used as an aid in removal of liner.

5.3.4.3 Detail procedure for application of film adhesive

(a) Roll the film adhesive into place so as to exclude air bubbles or other areas of entrapped air against skin metal. The adhesive may be tacked to metal parts by touching several spots with a clean, Teflon covered, hot tool such as a soldering iron or hot air gun. The temperature of the tacking tool cannot exceed 200° F.

(b) Trim the adhesive, leaving approximately 1/8 inch beyond the perimeter of the joining surface when permitted by the layout.

(c) More than one layer of adhesive film may be required in areas of mismatch. Additional layers will be limited by specifications on the drawing.

NOTE: If more than one piece of film is used on a part, butt splices will be used and gaps should not exceed 1/32 inch. Whenever possible, film splices should not be located closer than 1 inch to metal splicers.

(d) Parts must be bonded within the specified layup time as designated in the appropriate adhesive data sheet. If this exposure time is exceeded, the adhesive film must be removed and replaced with new film.

(e) Adhesive film that has been exposed to room temperature for a cumulative total exceeding the maximum time allowed by the adhesive data sheet must be discarded.

(f) Film adhesive must not be touched by bare hands. Wear clean gloves at all times while applying adhesive, peeling off protective liner and assembling of parts.

(g) Cutting tools, templates and tools used for laying out, cutting or handling of film adhesive should be vapor degreased or solvent cleaned as frequently as necessary to insure freedom from contamination.

5.3.5 Assembly of adhesively coated parts - Place parts in bonding fixture, if required, using suitable means for alignment. Insert thermocouples as specified in 5.3.6.4 (a).

5.3.6 Curing of adhesives

5.3.6.1 Autoclave bonding - When an autoclave is to be used in the curing of adhesives, the following procedure, or as directed by S&E-ME-M, is to be followed.

NOTE: It is understood that the following procedure is not all-inclusive and will not apply to all configurations. It is given in this document as a guide to aid in setting up the process for autoclave bonding.

(a) Place a bleeder material, such as a fiberglass cloth as a release sheet over the assembly and over edges and corners as required.

(b) Cover the sealed assembly with a vacuum blanket that is sealed to the fixture with sealant paste or equivalent.

(c) Check the sealed assembly for leaks by attaching a vacuum line, smoothing out any wrinkles that may form in the blanket or in the bleeder material, and apply a minimum of 14 inches of mercury. Close the vacuum line and check the indicating gage attached to the vacuum blanket. A vacuum drop of 2 inches or more per minute will indicate excessive leakage. Any leaks must be located, sealed, and rechecked.

(d) Place the sealed assembly in the autoclave, join the vacuum couplings, and determine the effectiveness of the seal.

(e) Check thermocouple junction with indicator leads.

(f) Close and seal autoclave. Apply autoclave pressure before temperature reaches 150° F. Release vacuum and vent assembly interior to atmosphere when the pressure in the autoclave reaches 15 psi and before the temperature exceeds 150° F.

NOTE: Additional vacuum may be applied if approved by S&E-ME-MM. Vacuum may be used during the complete cycle when nonfoaming type adhesives are used.

(g) Raise the temperature to the predetermined curing level at a suitable rate for the particular adhesive used. The autoclave may be preheated prior to loading, provided the pressure is applied before the bond line reaches 150° F.

(h) Maintain temperature and pressure for the time specified on the applicable data sheet.

(i) Cool parts to 150° F or less while still under bonding pressure. Cooling rate must be in accordance with that specified on the applicable adhesive data sheet.

5.3.6.2 Oven bonding - Follow similar techniques for oven bonding as specified for autoclave bonding. Repair bonding is sometimes accomplished in an oven.

5.3.6.3 Press bonding - Bonding in a platen press or unit tool will normally be limited to first stage metal-to-metal assemblies. When a platen press or unit tool is used to bond assemblies, suitable pressure distribution materials, such as liquid filled metal bladders, is used between the platen and the parts being bonded.

5.3.6.4 Curing time and temperature - The adhesive manufacturers recommendations should be carefully considered in establishing the curing conditions to be used in fabrication of the bonded assemblies.

(a) Curing temperature control - Unless otherwise specified, install at least one thermocouple in the bond line for each 10 square feet or fraction thereof, of peripheral length of the assembly, whichever is greater. The thermocouples used should be fabricated from premium grade iron-constantan wire Number 26 maximum, or equivalent, and be embedded in the adhesive bond or in the adhesive flash immediately adjacent to the edge of the

metal. Check each thermocouple for continuity at the time of its fabrication. Predetermine the rate of heating. Temperature differential at measuring points must not exceed 20° F upon reaching the curing temperature. The curing temperature of each adhesive bondline thermocouple installation must be continuously monitored and permanently recorded by an accurately calibrated temperature recorder during the cure cycle. The time the adhesive is above 100° F must be carefully controlled.

5.3.6.5 Bonding pressure - Adequate pressure must be maintained during the final cure of the adhesive bond to (1) obtain uniform adhesive bond thicknesses, (2) overcome internal pressure exerted by release of adhesive solvents, and (3) overcome the surface imperfections (within limits) between mating surfaces or the lack of flatness in the skin materials, (4) bonding pressure is also required to remove gases during the curing cycle. The pressure required for bonding is constant within the limits for any one adhesive, but may vary for different assemblies. Therefore; bonding pressure is established for each project as the project is designed and scheduled.

(a) Bonding pressure control - Maintain constant pressure on the entire bond area during the cure cycle. Apply pressure in a manner that will prevent lateral movement of the assembly parts or displacement of the adhesives. Production parts are to be cured at a pressure within ± 5 psi of the pressure used in qualifying the process. Curing pressure must be applied before the adhesive reaches a temperature of 150° F. Adhesive bonds should be cured under fluid pressure applied to the assembly and tools through a flexible blanket. The flexible blanket must be in direct contact with one surface of the assembly being bonded. Pressure may be applied through any material used between the part and the blanket that will uniformly transmit the fluid pressure. Wrinkles must be kept to a minimum. Bridging over step configurations is to be avoided.

(b) Loss of pressure - In the event pressure is lost before reaching 150° F, reseal the assembly and reheat.

5.3.7 Room-temperature-cure edge filler or potting compound - When room-temperature-cure edge filler or potting compound is to be used, curing temperatures, cure time, and cure pressures must be in accordance with the applicable adhesive data sheet.

5.3.8 Filling of cavities - For filling of cavities, inject compound with a Pyles gun, or equivalent. When potting for inserts, etc., is required, sufficient compound must be used to completely encapsulate the insert and fill the hole or cavity. Remove any excess compound immediately.

5.3.9 Finish Requirements - Finish the bonded assemblies in accordance with the requirements of applicable specifications.

6. PROCESS CONTROL TESTS

6.1 Preparation of Test Specimens - Production bonded assemblies should incorporate, where possible, extensions or coupons that can be cut from the assemblies after bonding that will be representative of the bonds in the assembly. Where extensions cannot be incorporated in the parts, fabricate two panels per Figure 9-3, one for peel tests and one for tensile tests (cuts into 6 specimens each). Bond these specimens at the same time, using the same equipment, and under the same conditions as for the assembly they represent.

6.2 Nondestructive Tests

6.2.1 Nondestructive Examination - Nondestructive evaluation must be made of the adhesive bond of each production part.

6.2.2 Visual Examination - All adhesive bonded structures must be visually examined immediately after manufacture to determine that they meet all requirements of the applicable engineering drawing with respect to configuration, dimensions, uniformity, and appearance. Bonded parts that show evidence of irregularities will be rejected or the defective areas will be repaired.

6.3 Destructive Tests

6.3.1 Metal-to-Metal Adhesives - Conduct room temperature tensile shear tests in accordance with Specification MMM-A-132 for aluminum. Use applicable Specifications for other metals. Five specimens are normally tested. See Paragraph 6.1.

6.3.2 Metal-to-Metal Peel Tests - This test is to be performed in accordance with this specification for determining conformance to the bonding requirements. Five specimens are usually required. See Paragraph 6.1.

6.4 Production Stoppage - When a production part has been rejected because of failure to meet test requirements, work on the production item must be stopped and not resumed until corrective measures are implemented.

7. NOTES

7.1 Intended Use - This Manufacturing Specification is intended for use, with applicable drawings, specifications and other referenced documents, by the Process Engineering Laboratory for fabrication of metal-to-metal, adhesively bonded, composite structures.

7.2 Safety - Exercise standard safety precautions with resins and chemical solutions.

7.3 Cleanliness - Cleanliness precautions must be exercised at all times for all bonded parts. Refer to M-ME-MPROC 005.5B-3 and M-ME-MPROC 005.5B-4, cleaning specifications.

7.4 Storage of Parts - Parts and components must be clean, free of contamination, wrapped in untreated Kraft paper, and stored in a clean, dry area.

MANUFACTURING SPECIFICATION
CLEANING FOR ADHESIVE BONDING

1. SCOPE

This specification covers the cleaning for Adhesive Bonding of Aluminum alloys.

2. DOCUMENTATION

2.1 Governmental

2.1.1 Federal Specifications

O-S-595A - Sodium Dichromate-Dihydrate

O-S-809A - Sulfuric Acid

O-T-634 - Trichloroethylene, Type II

CCC-C-440 - Cloth Cheese Cloth

MMM-A-132 - Adhesives, Heat Resistance, Air Frame Structural,
Metal-to-Metal

2.1.2 Military Specifications

MIL-C-3866A - Gloves, Cloth, Cotton, Knitted, Lightweight

MIL-C-7438C - Core Material, Aluminum, for Sandwich Construction

MIL-M-13999 - Methyl Ethyl Ketone, Reagent

MIL-P-17667A - Paper, Wrapping, Chemically Neutral

MIL-A-25463 - Adhesive, Metallic Structural Sandwich Construction

2.1.3 Standards

151A - Metals, Test Methods

165 - Adhesives, Test Methods

3.0 REQUIREMENTS

3.1 Facilities

- Central Missile Grade Air System

3.2 Chemicals

- Sodium Dichromate Dihydrate; Reagent Grade
- Sulfuric Acid; Reagent Grade
- MEK
- Trichloroethylene
- Turco 4215
- Acetone
- Demineralized water

3.3 Materials

- Kinwipes, Kraft Paper, Polyethylene 4 to 6 Mil-Film, Cotton Lint-Free Gloves
- Rubber Gloves, Face Shields, Aluminum Oxide Abrasive Cloth 250 to 400 mesh, and Masking Tape
- Clothing; Nylon and/or Disposable Smocks; Nylon and/or Disposable Caps as required in specified operations/and Lint Free, Cotton Gloves.

3.4 Equipment

- Vapor Degreasers
- Tanks - Tank Linings shall be compatible with chemicals used.

● Demineralized water spray equipment (spray nozzle) on every other tank (minimum).

- Drier - Forced air driers with filters built in or equivalent.

3.5 Chemical Controls

- Daily pH and conductivity checks on rinse water
- Twice a week check on 4215 Cleaner and Acid Etch

3.6 Chemical Requirements

● Trichlorethylene - Shall be inhibited and have a 188 ± 3 degrees F. Boiling Point.

● Alkaline Cleaner - The concentration of Turco 4215 shall be 12 ± 3 oz/gal at a temperature of 170 ± 5 degrees F.

● Acid Etch - The concentration shall be 40 ± 5 percent sulfuric acid, 5 ± 1 percent Sodium Dichromate and balance DI water at a temperature of 160 ± 5 degrees; or Forest Product Etch with concentration of 24 ± 2.4 percent Sulfuric Acid, 2.4 ± 0.24 percent Sodium Dichromate and balance DI water at a temperature of 160 ± 5 degrees F.

● Hot Water Rinse - Hot water rinse shall have a pH 6 to 8 with conductivity of a minimum of 50 000 OHMS at 160 ± 5 degrees F.

● Cold Water Rinse - Cold water rinse shall have a pH 6-8 with conductivity of a minimum of 50 000 OHMS at ambient temperature.

3.7 Cleanliness

● Clean Lint Free Gloves shall be worn at all times when handling clean parts.

- Cleanliness of Parts
- Parts shall show no water breaks when removing from rinse water.
- Dried parts shall show no spots or discolorations.
- Packaging - The clean dry parts shall be wrapped in brown paper or polyethylene film.
- Parts shall be recleaned if fabrication or priming has not occurred within 12 hours.

3.8 Solution - All solution shall be agitated during cleaning cycles.

4. PROCEDURE FOR CLEANING

4.1 Preclean - The parts shall be cleaned free of dirt, grease, oil or inks with a cloth moistened with Acetone or MEK. *

4.2 Degrease - The parts shall be vapor degreased until vapor ceases to condense, and/or spray parts while lowering.

4.3 Dry - Allow condensate to dry from parts before transferring to Alkaline Cleaner.

4.4 Alkaline Cleaner - The parts shall be immersed with agitation for 10 minutes in Turco 4215 at temperature of 170° F ($\pm 5^\circ$).

4.5 Rinse - Parts shall be spray rinsed when removing from Alkaline Cleaner to remove excess cleaner. Check for water breaks.

4.6 Hot Water Rinse - The parts shall be immersed in a hot water rinse for 5 minutes at a temperature of 160° F. Check water breaks for cleanliness.

4.7 Acid Etch - The parts shall be immersed in an agitated acid etch for 5 to 10 minutes at a temperature of 155° F ($\pm 5^\circ$).

* If light corrosion appears on any parts, the corrosion shall be removed by gently sanding with Aluminum Oxide Abrasive Cloth.

- 4.8 Rinse - Spray rinse the parts when removing from the Etch Solution until Etch Solution is removed.
- 4.9 Cold Water Rinse - The parts shall be rinsed in cold agitating water for 5 minutes at ambient temperature.
- 4.9.1 Spot check part with pH paper to insure that water on the parts is not acidic.
- 4.10 Cleanliness - There shall be no water breaks on the parts when removed from cold water rinse.
- 4.11 Dry - These parts shall be allowed to dry for several minutes before placing in drying oven.
- 4.12 Forced Air Dry - The parts shall be placed in a forced air circulating oven 30 minutes at 150° F ($\pm 5^\circ$)
- 4.13 Cool - Parts shall be allowed to cool sufficiently before handling.
- 4.14 Cleanliness - The panels shall show no discoloration after drying.
- 4.15 Package - Parts shall be wrapped with brown Kraft paper or with Polyethylene.
- 4.15.1 Parts shall be primed at this time if applicable.
- 4.16 Recleaning
- 4.16.1 Parts shall be recleaned if parts have exceeded 12 hours before use or have been contaminated.
- 4.17 Cleaning Aluminum Core Materials - Core shall be vapor degreased only.

5. QUALITY ASSURANCE

5.1 Inspection points

- 5.1.1 Refer to paragraph 2.

- 5. 1. 2 Refer to paragraph 3. 1.
- 5. 1. 3 Refer to paragraph 3. 5.
- 5. 1. 4 Refer to paragraph 3. 7.
- 5. 1. 5 Refer to paragraph 4. 9. 1.
- 5. 1. 6 Refer to paragraph 4. 10.
- 5. 1. 7 Refer to paragraph 4. 14.

MANUFACTURING PROCESS FOR PREPARING
SUBSTRATES FOR ADHESIVE BONDING

1. SCOPE

1.1 Scope

This specification establishes the general procedures, preparation of cleaning solutions, and material requirements for pre-bond cleaning of adhesive bonded structures.

Non-metals and metals now being bonded are included, with the exception of aluminum which is contained in M-ME-MPROC-005.5B-3.

1.2 Applicability

The provisions of this specification are applicable to selected metal and non-metals composite structure substrates bonded for or by the Process Engineering Laboratory.

1.3 Restrictions

No other materials or processes may be used as alternates or equivalents to those required by this document without prior approval of the S&E-PT-M and S&E-PT-D Divisions.

2. APPLICABLE DOCUMENTS

2.1 Governmental

The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the latest revision shall apply.

SPECIFICATIONS

Federal or Military

(Chemicals)

O-A-51C

Acetone

MS-36039

Chromic Acid

MS-36039

Chromium Trioxide

<u>Federal or Military</u>	<u>(Chemicals)</u>
O-E-760D	Ethyl Alcohol (denatured)
MS-36043	Hydrochloric Acid (37% Solution)
MS-36044	Hydrofluoric Acid (1.18 Sp. G. or 49% Solution).
MIL-H-11357	Hydrogen Peroxide (30% Solution)
O-M-232B	Methyl Alcohol
TT-M-261B	Methyl Ethyl Ketone
TT-N-95A	Naphtha, Aliphatic
O-O-690	Oxalic Acid
O-T-634A	Perchloroethylene
O-P-313	Phosphoric Acid (87% Solution)
O-P-559	Potassium Dichromate
O-S-595A	Sodium Dichromate
MS-36037	Sodium Hydroxide (For Cleaning Purposes)
MS-36229	Sodium Metasilicate
O-S-0037	Sodium Pyrophosphate
O-S-809A	Sulfuric Acid
MS-36052	Sulfuric Acid (1.84 Sp. G. or 96% Solution)
MIL-M-13999	Methyl Ethyl Ketone, Reagent
MIL-T-10451A	Tetrasodium Phosphate

Federal or Military(Chemicals)

O-T-634A

Trichloroethylene

JAN-T-171

Toluene

MM-A-132

Adhesives, Heat Resistance, Air Frame
StructuralFederal or Military(Miscellaneous Supplies)

CCC-C-440

Cloth, Cheesecloth Cotton, Bleached
and Unbleached

AA-G-421A

Gloves, Rubber, Surgeons

MIL-G-3866

Gloves, Cloth, Cotton, Knitted, Light-
Weight

MIL-G-21380A

Grit Blast

MIL-P-17667

Paper, Wrapping, Chemically Neutral

Federal or Military(Substrate Materials)

MIL-B-21531

Beryllium

QQ-S-741B#1

Carbon Steel

MIL-P-55010A

Mylar

MIL-R-9300

Reinforced Plastic

MIL-P-22241A

Teflon

MIL-T-904GE

Titanium

STANDARDS

Federal

FED-STD-151A Metals, Test Methods.

Federal

FED-STD-165 Adhesives, Test Methods.

FED-STD-175 Adhesives, Methods of Testing.

STANDARDS

George C. Marshall Space Flight Center

MS-150.0 Vapor Degreasing

50M02112 Apollo Telescope Mount System Cleanliness Requirements

NOTE: In all cases adhesive vendors brochures for adhesives and primers from the Composite Structures Manual should be consulted.

3. EQUIPMENT AND FACILITIES

3.1 Equipment

- Vapor Degreasers
- Tanks - Tank linings shall be compatible with chemicals used.
- Demineralized water spray equipment (spray nozzles).
- Drier - Forced air driers with filters.
- Air-water Blast Equipment and Aluminum-Oxide Blast Equipment.
- Racks, Insulated

3.2 Facilities

- Surface Treat Facilities - Bldg. 4760 and 4707
- Laboratory Facilities - Bldg. 4760

4. MATERIAL REQUIREMENTS

4.1 Materials

- Paper, Kraft (MIL-P-1766)
- Gloves, white, lint-free (MIL-G-3866)
- MEK, Methyl Alcohol, or Acetone (TT-M-261)
- Missile Grade Air
- Aluminum Oxide 200 Mesh
- pH paper range 1 to 14
- Cloths, clean, white, lint-free (CCC-C-440)
- Clothing; Nylon and/or Disposable Smocks; Nylon and/or Disposable Caps as required in specified operations and Lint Free Gloves.

NOTE: The chemicals used for cleaning of a specified material are designated in the respective cleaning procedure for that material.

5. GENERAL INFORMATION AND TECHNIQUES

5.1 General Requirements

A clean, dry, grease-free surface is required for optimum adhesive bond strength. This surface condition is obtained by prebond cleaning. The prebond process generally consists of an initial degreasing step, the chemical etching of the substrate, thorough rinsing and then drying. In some cases the mechanical abrading of the surface to be bonded will suffice.

In many cases special chemical treatment will produce further significant improvements in bond strength and where the maximum strength is required, careful attention must be given to the surface pretreatment. Surface pretreatment involves one of the following three procedures which are listed in general order of merit:

- Degrease and chemically pretreat.

- Degrease, abrade and degrease again.
- Degrease only.

5.2 Degreasing

The initial degreasing step may be accomplished by using either a liquid or a hot vapor. In both cases the degreasing process is continued until the degreasing material does not bead upon the substrate but gives uniform wetness across the entire area to be bonded.

5.2.1 Degreasing Procedures

The preferred method for metal surfaces is as follows:

(a) Suspend in a stabilized trichloroethylene or perchlorethylene vapor degreasing bath (except titanium) for about 30 seconds, (MS-150). Check the bath frequently for accumulated contaminants.

OR: Where a degreasing unit is not available.

(b) Wipe the surface with clean white cotton cloth (MIL-6-3866) or (CCC-C-440) or absorbent cotton containing trichloroethylene or perchlorethylene and allow to stand for a minute or two to permit complete evaporation. Note that the cloth should not be too wet so that application to the surface will merely dissolve the grease but not absorb it into the fabric. These solvents are toxic in both liquid and vapor form. Rubber gloves should be worn and the place of work should be well ventilated. No smoking should be allowed. Change the cloth frequently.

Some metals may be degreased with detergent solution or hot alkali and reference is made to this under specific material treatments in the latter part of this document.

5.3 Chemical Pretreatments

To obtain optimum strength joints involving metals, a chemical or electrolytic pretreatment is normally recommended. Such treatments can increase the bond strength by as much as 300 percent. This type of pretreatment either etches the surface of the metal, or deposits complex inorganic inhibiting coatings. These special procedures are listed under the individual materials in the following paragraphs.

5.3.1 Etching

The chemical etching not only removes surface material but also serves to build up a polar film which forms a strong secondary linkage with the polar adhesives.

All etching solutions should be prepared in glass porcelain, polyethylene, polypropylene or "Teflon" laboratory ware and stirred with rods of the same material. "Teflon" should be used for solutions containing hydrofluoric acid or flourides. Solutions in plastic trays can be heated by immersion in hot water baths, while hot plates or infra-red heaters may be employed for glass and porcelain trays.

Because of the likelihood of contamination of the metal surface during storage, it is desirable to use the etched metals as soon as possible after treatment. If storage is unavoidable, the metal should be covered with lint-free tissue paper and kept tightly wrapped or in an airtight container to minimize contamination.

5.4 Rinsing

During the final rinse, the cleaned surface should be observed for water breaks. Should water breaks be observed, the cleaning process should be repeated. A water contact angle test as defined in Military Specification MIL-A-9067C, Item 6.1.5 may be used to determine the degree of cleanliness of the cleaned dry substrate. This method consists of placing a drop of distilled water on the flat, cleaned, dry surface, and using a microscope with a cross hair in the rotating eye piece and a prism on the objective end. A low contact angle of 10 degrees or less indicates satisfactory preparation for adhesive bonding.

The true test for a well cleaned surface is the ease with which it is wetted by water which would form beads of droplets on unclean surfaces and a continuous film on a clean surface.

5.5 Abrasion Procedures

Lightly abraded surfaces provide superior adhesion to polished surfaces, due to their increased complexity and surface area. Abrasion should always be followed by a second degreasing treatment to remove abrasion contaminants.

Surface deposits such as oxide films, tarnish, rust, mill-scale, etc., are best removed by grit-blasting with fine grit. However, this can only be employed on structures of sufficient thickness to prevent distortion. Vapor "honing" (which is similar to grit-blasting, but employs high velocity water or steam in place of air) may sometimes be used.

Stains and oxides on metal surfaces may be removed by light sanding with 200-grit emery wet/dry paper.

Plastics may first require removal of a wax or other mold release agent by means of a specified solvent. This material often has the surface roughened by a fine grit emery paper.

Mark all areas not to be sanded or dissimilar materials which could be damaged by the cleaning solutions. All holes, through or dead end, shall be plugged.

Handling of the cleaned parts must be done by personnel wearing clean, lint-free gloves. If cleaned parts require transportation or short time storage they shall be protected by wrapping with clean kraft paper, meeting requirements of MIL-P-1766.

6. SAFETY

6.1 Precautions

All applicable safety rules should be observed when preparing the chemical solutions required to prebond clean the substrates, and when performing the actual cleaning operations. These safety rules may be obtained from the Safety Department.

Preparation and handling of cleaning solutions demands the exercise of rigid safety precautions and all contact with the body must be avoided.

Utilize all standard safety requirements by wearing safety acid-proof clothing, hoods, boots, gauntlet gloves and face masks. Work shall be performed in a well ventilated area or while wearing air supply masks.

When preparing solutions containing concentrated acid, it is most important that the acid be added to the water, since violent "spitting" occurs if water is added to the acid.

Care must be taken in the preparation of the chemical solution, since incorrect proportioning may lead to bond strengths inferior to those that would have been obtained if there had been no chemical pretreatment at all. It should also be noted that many of the materials recommended are strong acids or bases and are highly reactive on the skin; therefore, face masks, goggles, slip-proof neoprene gloves, rubber aprons and rubber boots should be provided.

7. PROCESSING OF METALS

7.1 Beryllium

- (a) Prepare caustic soda solution:

Sodium hydroxide	20 parts by weight
Distilled or deionized water	80 parts by weight

Dissolve 20 parts of sodium hydroxide in an equal weight of water followed by addition of the remainder of the water.

- (b) Degrease beryllium parts in Freon vapor bath and air dry per MS-150.0. Maintain solvent level above the steam coils. Adjust the automatic controls to 169° F ($\pm 5^\circ$) so that the top of the vapor zone is maintained near the top of the cooling coils.

- (c) Dip in the caustic solution for 3 minutes at 180° F ($\pm 5^\circ$).

- (d) Remove the parts from cleaning solution tank and wash in deionized water. If upon removal from the rinse tank water breaks occur, repeat the entire cleaning procedure.

- (e) Dry the part in a forced air drying oven, equipped with air filters at a controlled temperature at 290° F ($\pm 5^\circ$) for 30 (± 5) minutes.

7.2 Copper and its alloys (Brass, Bronze)

- (a) Prepare ferric chloride.

42 percent aqueous ferric chloride solution	15 parts by weight
Concentrated nitric acid, reagent grade	30 parts by weight
Distilled Water	197 parts by weight

- (b) Degrease copper parts in trichloroethylene vapor bath and air dry per MS-150-0.
- (c) Dip in ferric chloride solution for 1 to 2 minutes at room temperature.
- (d) Rinse well with distilled or deionized water.
- (e) Dry immediately with filtered air stream at room temperature, (use of hot air may strain the surface).

7.3 Magnesium and Magnesium Alloys

7.3.1 Degrease per MS-150.0 and etch using the following solutions:

Solution "A"

Sodium Metasilicate	55 parts/weight
Tetrasodium Pyrophosphate	35 parts/weight
"Nacconol NR" (Allied Chem. Co.)	5 parts/weight
Sodium Hydroxide	15 parts/weight
Distilled water	2,400 parts/weight

Add the sodium metasilicate, tetrasodium pyrophosphate, sodium hydroxide and "Nacconal NR" slowly to the agitated deionized water. Continue stirring, and for future use it should be agitated prior to use to insure adequate mixing.

Solution "B"

Chromium Troxide	13 parts/weight
Calcium Nitrate (Commerical Grade)	1 part/weight
Distilled Water	73 parts/weight

7.3.2 Treat the magnesium as follows:

- (a) Degrease per MS-150.0
- (b) Immerse in Solution "A" for 5 to 10 minutes at 165-185° F.
- (c) Rinse thoroughly with running, ambient deionized or distilled water.

- (d) Immerse in Solution "B" for 5 to 15 minutes at room temperature.
- (e) Rinse thoroughly with ambient distilled or deionized water.
- (f) Dry in an oven for 15 to 20 minutes at 175 to 200°F.
- (g) Cool and apply adhesive.

7.4 Steel (Stainless)

7.4.1 Degrease per MS-150.0. Remove any surface deposits with non-metallic agents (e. g., alumina grit paper) and degrease again. For the final degreasing, the following procedure is recommended:

- (a) Degrease with trichloroethylene per MS-150.0.
- (b) Immerse in the following alkaline detergent solution for 10 minutes at 160 to 180 °F. (See paragraph 7.3.1 for mixing instructions.)

Sodium Metasilicate	3 parts/weight
Tetrasodium Pyrophosphate	1.5 parts/weight
Sodium Hydroxide	1.5 parts/weight
"Nacconol NR" (Allied Chemical Co.)	0.5 parts/weight
Distilled Water	134.0 parts/weight

(c) Rinse thoroughly with ambient running, tap water, followed by distilled or deionized water.

(d) Dry in an oven at 200°F for 15-20 minutes. Where maximum heat resistance or peel strength is required, one of the following two treatments should be employed.

7.4.1.1 For maximum heat resistance, prepare the following solution:

Oxalic Acid	1 part/weight
Concentrated Sulfuric Acid (S.G.I. 86)	1 part/weight
Distilled Water	8 parts/water

Place the oxalic acid in the water and stir until a saturated solution is obtained. Then, add the sulfuric acid SLOWLY WITH CONSTANT STIRRING (CARE!). The oxalic acid will dissolve completely at the operating temperature shown below. Treat the steel as follows:

- (a) Degrease with trichloroethylene per MS-150.0
- (b) Immerse in the alkaline detergent solution already described for 10 minutes at 160-180°F).
- (c) Rinse thoroughly with ambient tap water.
- (d) Immerse in the oxalic-sulfuric acid solution for 10 minutes at 180 to 195°F.
- (e) Remove black deposit by scrubbing the metal with a clean, stiff brush under ambient tap water.
- (f) Rinse in distilled or deionized water at ambient temperature.
- (g) Dry in an oven for 10 to 15 minutes at 200°F.

7.4.1.2 For maximum peel strength, prepare the following sulfuric acid solution:

Sodium Dichromate	3.5 parts/weight
Distilled Water (Or deionized)	3.5 parts/weight
Concentrated Sulfuric Acid (S. G. I. 86)	200.0 parts/weight

Place the sodium dichromate in the deionized water and stir until a saturated solution is obtained. Add the concentrated sulfuric acid slowly with constant stirring until the sodium dichromate dissolves. For future use it should be agitated prior to and during etching.

Treat the steel as follows:

- (a) Degrease with trichloroethylene per MS-150.0.
- (b) Immerse in the alkaline detergent solution already described, for 10 minutes at 160 to 180°F.
- (c) Rinse thoroughly with clean, ambient water.
- (d) Immerse in the sulfuric acid solution for 15 minutes at 140 to 160°F.
- (e) Remove and scrub the steel with a clean, stiff bristle brush under clean, ambient, running water.

- (f) Rinse in distilled or deionized water.
- (g) Dry in an oven for 10 to 15 minutes at 200° F.

7.5 Steel (Mild) and Ferrous Alloys other than Stainless Steel

7.5.1 Degrease per MS-150.0 and etch in the following phosphoric acid solution:

Concentrated Orthophosphoric Acid (S. G. I. 73)	50 parts/weight
Ethyl Alcohol (denatured)	50 parts/weight

Treat the metal as follows:

- (a) Degrease (MS-150.0)
- (b) Immerse in the phosphoric acid solution for 10 minutes at 400°F.
- (c) Remove black deposit by scrubbing the metal with a clean, stiff brush under distilled or deionized ambient water.
- (d) Heat metal for 1 hour at 250° F.

7.6 Titanium and Titanium Alloys

7.6.1 Degrease with Methyl Ethyl Ketone. Remove any surface deposit with non-metallic agents, e.g. alumina grit paper. Etch with the following acid solution:

Sodium Fluoride	10 parts/weight
Chromium Trioxide	5 parts/weight
Distilled Water	250 parts/weight
Concentrated Sulfuric Acid (S. G. I. 86)	50 parts/weight

Dissolve the sodium fluoride and chromium trioxide in the water and add the sulfuric acid SLOWLY WITH CONSTANT STIRRING (CARE!). The solution should be contained in and stirred with equipment made of Teflon.

Treat the titanium as follows:

- (a) Degrease with MEK.
- (b) Immerse in the alkaline detergent solution recommended for stainless steel for 10 minutes at 160 - 180° F.
- (c) Rinse thoroughly with ambient tap water.
- (d) Immerse in the above acid solution for 5 - 10 minutes at room temperature.
- (e) Rinse in ambient, distilled or deionized water.
- (f) Dry in an oven for 10 - 15 minutes at 160 - 180° F.

7.6.2 Preparing Titanium for Bonding with Pasa-Jell 107 and 107M. (Alternate Method).

7.6.2.1 Description

Pasa-Jell 107 and Pasa-Jell 107M are blends of mineral acids, activators, and inhibitors. Pasa-Jell 107 is inorganically thickened to permit application in localized areas. Pasa-Jell 107M does not contain the inorganic thickener permitting immersion of large surfaces. Each Pasa-Jell is designed to provide a balanced ratio of components which will maintain effectiveness of etch rate and inhibit embrittlement so long as the total acid content is maintained above 20 percent.

Improvement of bond strength is based on deposition of a conversion coating which provides a receptive surface for adhesion.

Pasa-Jell 107 and Pasa-Jell 107M chemically cleans the titanium alloy surface to provide improved chemical bonding of the adhesive. Pasa-Jell 107 and Pasa-Jell 107M leave a clean surface without a trace of smut.

Pasa-Jell 107 and Pasa-Jell-M are formulated specifically for titanium alloys and a different formulation is required for pure titanium.

The etch life of Pasa-Jell 107-M is approximately 500 to 1000 square feet of surface per gallon.

7.6.2.2 Physical Properties (Typical)

Color	Red
Total acid content	
Pasa-Jell 107	Above 22 Percent
Pasa-Jell 107-M	Above 22 Percent
Weight per gallon	
Pasa-Jell 107	9.5 pounds
Pasa-Jell 107-M	9.0 pounds
Consistency	
Pasa-Jell 107	Thixotropic gel
Pasa-Jell 107-M	Thin liquid

7.6.2.3 Application Instructions

For optimum results the titanium alloy surface should be solvent degreased with a non-chlorinated solvent followed by either a wet or preferably dry blast with a fine grit, 250 to 320, aluminum oxide to remove all corrosion products and to condition the metal surface. Where blasting is not practical, the surface should be sanded lightly with aluminum oxide coated abrasives. Satisfactory results may be obtained with the use of alkaline cleaners.

Apply a layer of Pasa-Jell 107 to localized areas with a polyethylene, polypropylene or fluorocarbon bristle brush immediately following the abrasive treatment and allow to stand for 10 to 15 minutes after which time the Pasa-Jell should be rinsed off with cool, tap water and allowed to dry at room temperature.

For immersion treatment of titanium alloy skins, use Pasa-Jell 107-M because of its lower viscosity.

NOTE: Tank linings for Pasa-Jells should be made of polyethylene or polyvinyl chloride. Polyvinyl chloride linings should be pretreated with a 50 percent nitric acid solution for 1 hour at room temperature to remove surface plasticizers before placing the tank in service.

It is not advisable to add free hydrofluoric acid to maintain the etch rate of the solution because it will lower the bond value. Ferric and ferrous ion contamination should be avoided because it reduces the activity of the Pasa-Jell.

Pasa-Jell 107-M, as supplied, is approximately 22 percent total acids but can be maintained at a higher acid content depending upon the desired rate of etch. Pasa-Jell 107-M should always be maintained at 20 percent or higher by the addition of Pasa-Jell 107-C concentrate.

Maintenance of surface tension of the Pasa-Jell 107-M tank solution below 55 dynes per centimeter is important to obtain proper performance and can be accomplished by the addition of Semco Surfactant No. 7. Recirculation of the tank solution by pumping is preferable to air agitation.

It is important that the part and the Pasa-Jell 107-M tank solution be held at a maximum temperature of 100°F. This also applies to any tap water rinsing. Treated parts should be dried with cool air prior to priming.

The time limit for application of primer after rinsing should be held to a minimum and preferably not in excess of 1 hour because there is a decay rate of the surface activity and maximum benefit can be derived only by priming the surface as soon after the Pasa-Jell treatment as possible.

In the event detergent cleaning and acid pickling are employed prior to the Pasa-Jell treatment, the following precautions should be observed:

- (a) Make sure the titanium alloy skin is clean before heat treatment.
- (b) Seizure of detergent solution on titanium alloy surface should be avoided. Keep the part being cleaned from drying. High pressure spray rinse immediately upon removal of part from the detergent tank is recommended.
- (c) Check parts as removed from the acid pickle solution to make sure a uniform etch has been obtained. If nonuniformity of etch is observed, immerse the part for a longer time period or repeat the entire cycle.
- (d) Should it be necessary to recycle the part after the Pasa-Jell treatment, the conversion coating first should be removed by immersion in a nitrichydrofluoric acid pickle solution and the above steps repeated.

Pasa-Jell is supplied by the Products Research & Chemical Corporation. Corporation Offices & Western Manufacturing Division, 2919 Empire Avenue, Burbank, California 95104, (213) 849-3992 - Eastern Sales & Manufacturing Division, 410 Jersey Avenue, Gloucester City, N. J. 08030, (609) 456-5700.

7.6.2.4 Safety Precautions

DANGER: EXTREMELY HAZARDOUS LIQUID AND VAPORS

Causes irritation of skin, eyes, nose and throat. Avoid contact with skin, eyes and clothing and avoid breathing vapors. Store in a cool, dry area in tightly closed containers. In case of spillage, drench with large quantities of water.

Pasa-Jell 107 and Pasa-Jell 107-M have been proven to be safe materials to handle when safety and handling precautions are followed. Avoid contact with skin and clothing. Wear rubber gloves, face shield and respirator protection. Avoid breathing vapors. Always work in a well ventilated area.

7.7 Metal Honeycombs

Normally honeycombs of aluminum, stainless steel or titanium are furnished clean. However, for optimum adhesion, if oil or grease contamination is suspected treat as follows:

- (a) Vacuum clean
- (b) Degrease per MS-150.0 (Use MEK for titanium).
- (c) Dry in air at R.T. for 2 to 3 hours or in oven for 1 hour at 150-200° F.
- (d) Examine for cleanliness as the degreasing operation may have to be repeated.

8. NON-METALS

8.1 Ceramics (unglazed)(e.g. porcelain, glazed china)

Degrease. Remove glaze with grit-blast or emery paper. Degrease again with MEK. Allow to stand for a few minutes to complete evaporation of the solvent.

8.2 Glass and Quartz

Degrease per MS-150-0, and dry at 210° F, and etch in the following solution:

Chromium Trioxide	1 part/weight
Distilled Water	4 parts/weight

Immerse the glass for 10 to 15 minutes at room temperature. Wash well with distilled water and dry at 210°F for 30 minutes. Apply the adhesive while glass is hot for optimum strength.

NOTE: Glass for optical purposes must not be exposed to etches of any kind. The glass should be cleaned in a detergent bath, preferably one which is ultrasonically agitated, followed by thorough rinsing. Drying temperatures should be kept below 100 to 125°F.

8.3 Plastics

Many plastics are coated with a wax for improved appearance or mold release agent. These substances must be removed with a solvent or detergent solution. Depending upon the plastic, either acetone or methyl alcohol are recommended for use as solvents.

NOTE: Examples of commercial detergents are "Oakite" (Oakite Products, Inc., 19 Rector Street, New York 6, N. Y.) "Sprex" (The DuBois Co., 1120 W. Front, Cincinnati, Ohio).

8.4 Melamine

Formaldehyde Plastics (e.g., "Formica") - Degrease with a cloth containing acetone or MEK. Abrade the surface with medium-grit emery paper. Degrease again with acetone or MEK.

8.5 Nylon

Degrease with a cloth containing acetone or MEK. Roughen with medium-grit emery paper. Degrease again.

8.6 Mylar

Polyethylene Tetrathalate and Linear Polyesters Degrease with a cloth containing methyl alcohol. Abrade with medium-grit emery paper and degrease again, OR preferably treat as follows:

- (a) Degrease with methyl alcohol

(b) Immerse in a 20 percent by weight sodium hydroxide solution for 2 to 10 minutes at 160-200° F.

(c) Wash thoroughly with ambient, distilled or deionized water.

(d) Dry with hot air (160 - 180° F).

8.7 Polycarbonate, Polymethyletracrylate (Acrylic), Polystyrene

Degrease with a cloth containing methyl alcohol. Abrade with aluminum oxide 200-mesh paper and degrease again with methyl alcohol.

8.8 Phenolic Resins, Polyester Resins, Polyurethane resins

Degrease with lint free cloth containing acetone or MEK. Abrade with aluminum oxide 200-mesh paper. Degrease again with MEK.

8.9 Polymonochlorotrifluoroethylene (e.g. "Kel-F"), Polytetrafluoroethylene (P.T.F.E.) (e.g. "Teflon), Polyvinylfluoride (P.V.F.)

Prepare the following solution:

Sodium Metal	23 g.
Napthalene	128 g.
Tetrahydrofuran (Technical Grade)	1 liter

Place the tetrahydrofuran in a 2 liter glass flask, dissolve the napthalene and then add the sodium (CARE!) in the form of 1/4 to 1/2 inch cubes, dissolving 1 cube at a time with stirring.

The resulting solution is very water sensitive and the flask must be equipped with a drying tube outlet and a mercury sealed stirrer.

Allow the solution to stand overnight at room temperature and then stir until a buish-black liquid results. This must be stored in glass stoppered bottles when not in use, also a glass container is required while in use. An exhaust system for ventilation should be provided when the bath is in use. Prepare the plastic as follows:

(a) Degrease with acetone or MEK using a lint-free cloth.

- ture. (b) Immerse in the above solution for 15 minutes at room temperature.
- (c) Wash in acetone or MEK.
- (d) Wash in ambient, distilled or deionized water.

9. OTHER METALS

9.1 Nickel and Cobalt Base Alloys

- René 41
- Haynes 25 (L 605)
- Inconel 718
- Hastelloy X
- Inconel 625
- Inconel X750

9.2 Preparation for Bonding

9.2.1 Vapor degrease per MS-150.0 or emulsion clean and rinse parts.

NOTE 1 Allow the solvent to evaporate from the part before further processing.

NOTE 2 After degreasing, parts will be handled with clean white gloves.

9.2.2 Alkaline Clean

- (a) Soak in Turco Vitroclean (or equivalent) for a minimum of 10 minutes.
- (b) Solution 7 to 10 oz/gal., Temperature $190 \pm 10^{\circ}\text{F}$, Rack according to accepted procedures.
- (c) Scrub soils and markings not removed using clean, white cloths or approved soft bristle brush. If an abrasive is needed, use Pumice Grade FF or Wyandotte F1013 (or equivalent).

(d) Spray rinse with deionized water to remove excess cleaner and/or abrasive.

(e) Immersion rinse in deionized water. Use a minimum temperature of 110°F if parts are to be adhesively bonded.

(f) Inspect for water breaks, if necessary repeat the cleaning process.

(g) Dry with Missile Grade Air or dry with clean absorbent cloth.

(h) Wrap parts in Kraft paper to assure maintenance of surface cleanliness.

10. REFRACTORY ALLOYS

10.1 Columbium (Niobium)

- C103
- CB-752
- C-129Y
- WC-3015

10.2 Tantalum

- Ta-10W
- T-111
- T-222

10.3 Preparation for Bonding

10.3.1 Vapor Degrease with Trichloroethylene Per MS-150.0.

- (a) Rack parts according to standard procedures.
- (b) Hold parts in vapor zone until condensation stops.
- (c) Remove parts slowly.

10.3.2

Alkaline Clean

(a) Soak in Turco Vitroclean (or equivalent) for minimum of 10 minutes. Solution: 8 to 10 oz/gal. - Tem: $190 \pm 10^\circ \text{F}$.

NOTE: Rack according to accepted procedures.

(b) Scrub soils and markings not removed in above process (using clean white gloves) with approved soft bristle brush.

(c) Spray rinse to remove excess cleaner.

(d) Immersion rinse in deionized water, minimum temperature of 110°F if parts are to be adhesively bonded.

(e) Inspect for water breaks; if necessary repeat alkaline clean.

(f) Dry with Missile Grade Air or dry with clean, absorbent, cloth.

(g) Wrap parts in Kraft paper to assure maintenance of surface cleanliness.

11. QUALITY ASSURANCE

11.1 Requirements

(a) After processing, surfaces shall be free from all surface contamination.

(b) Parts which have been pitted or etched beyond minimum surface dimensions and surface finish requirements as specified in the drawings or specifications will be rejected.

12. HANDLING AND STORAGE

12.1 Requirements

(a) All handling of parts after cleaning must be controlled to prevent contamination.

(b) Careful handling throughout all operations is mandatory.

SECTION X

SURFACE TREATMENT / PAINTING

Manufacturing process data and specifications for surface treatment, painting, silkscreening, etc., that have been successfully used at MSFC are contained in this section of the manual.

- Manufacturing Process Data - Paint Removal from S-IC/S-II

Test Container

- Zinc Chromate Priming of the Fuel and Oxidizer Container

Bulkheads

- Zinc Chromate Priming of S-IC Components

- Application of S-13 Thermal Coating

- Manufacturing Process Data - Application of S-13G Thermal

Control Coating

- Zinc Chromate Priming of all S-IC Weld Areas

- Removal and Repair of Anodic Coating on Parts to be

Welded

- Process Control and Operating Procedures for Cadmium

Plating of Space Vehicle Parts

- Process Control and Operating Procedures for Nickel Sul-

famate Plating of Space Vehicle Parts.

- Manufacturing Specification - for the Painting of Space

Launch Vehicle Surfaces

- Manufacturing Specification - for Silk-Screen Reproductions

- Manufacturing Process - for Surface Treatment of Weld

Areas of the S-IC Bulkhead Test Fuel Tank

- Manufacturing Process - for the Application of Dye Pene-

trant to Tank Surfaces (Non-LOX Wetted) for S-IC Vehicle

MANUFACTURING PROCESS DATA FOR
PAINT REMOVAL FROM THE S-1C/S-II TEST CONTAINER

1. SCOPE

1.1 This Manufacturing Process Data outlines the procedure recommended for removing the paint and primer from this tank. A mechanical cleaning process is included for reducing the oxide film prior to further cleaning.

2. EQUIPMENT AND MATERIAL

2.1 Equipment

- Scaffolding
- Highlife vehicles
- Paint brushes
- Scrub brushes
- Stiff fiber long handled push brooms
- Water hoses
- Compressed air supply
- Nozzles for air and water for flushing operations

2.2 Material

- Cee Bee A 202
- Turco 5003
- MEK (Methyl Ethyl Ketone) Solvent
- Clean cotton rags

2.2 Materials (continued)

- 95-percent ethyl alcohol, technical grade
- Toluene, reagent grade
- Cotton swab
- Kerosene
- Dacron cloth

3. PROCEDURE

3.1 Surface Preparation

Purpose - to promote adhesion of the temperature control paint.

3.1.1 Aluminum

Parts that are constructed of aluminum alloys shall be chemically treated by only one of the three following methods.

3.1.1.1 Preclean in accordance with MS 150.2A, Type 1

3.1.1.2 Vapor degrease as specified in MS 150.0

A. Etch in a 5-percent solution of sodium hydroxide (NaOH) for 3 minutes at room temperature.

B. Rinse with deionized water.

C. Desmut in a 15-percent solution of Nitric Acid for 20 to 30 minutes.

D. Rinse with deionized water and dry.

E. Rinse with Anhydrous Alcohol and let dry.

NOTE: Parts or assemblies adhesively bonded or that include polymeric or organic materials in their construction shall not be cleaned as in paragraph 3.1.1.2

3. 2 Masking

Surfaces that are to be free of paint shall be masked with pressure sensitive tape. Parts that are subject to damage by tape removal or contaminated by tape adhesive shall be so noted. Contact with temperature control surfaces shall be kept to an absolute minimum and the operator's hands shall be clean since handling of the parts with special gloves may not be feasible during masking operations.

3. 2. 1 Where contours exist, pulling of tape shall be avoided. Demasking shall be performed carefully three (3) to six (6) hours after the final coat of paint has been applied to avoid starting of pulling stresses in the paint coating. Any remaining adhesive shall be removed cautiously avoiding contact with or contamination of the painted surface with solvents. To remove adhesive residue, first wipe the surface carefully with a swab of cotton or dacron saturated with kerosene or solvent, follow with a toluene wipe, and allow to dry.

3. 3 Priming

3. 3. 1 Use General Electric Primer SS-4044. Spray apply a thin film (0. 2 to 0. 6 mil) with a Binks Model No. 18 spray gun with suction feed. For the primer a Paasche AU70 air brush with ARF-1 tip may be needed to obtain desired coverage.

3. 3. 2 Allow primer to dry for 1 hour at room temperature before further processing. The time between priming and application of first coat of S-13G paint must not exceed 24 hours.

3. 3. 3 Lower plastic shroud over unit during drying time for primer, in a manner that will not prevent solvent evaporation.

NOTE: (1) The S-13G coating will not adhere to an unprimed surface. Make certain that the entire unit is covered with primer.

(2) All work with the S-13G coating is done under clean room conditions of temperature, humidity, and contamination controls.

(a) The spray booth module shall be provided with an air filtration system.

(b) The paint facility shall be capable of maintaining the temperature at $75 \pm 10^{\circ}$ F. and a relative humidity of less than 50-percent.

(3) The velocity of the air flow at the spray table in the paint booth shall be within 100 to 150 feet per minute.

3.4 Mixing Instructions for S-13G Coatings

Mix the S-13G paint in one pint batches, in a glass beaker, by hand, 15 minutes prior to use. The first step in the mixing process is to check and adjust the viscosity to 15 - 17 seconds with a No. 4 Ford Cup using Toluene as a thinner. The SRC-05 catalyst is mixed separately with Toluene on a 1:10 ratio, respectively. The next step is to mix 7 grams of catalyst to 690 grams of paint and stir thoroughly. The final step includes straining twice through a nylon sieve to remove any particles that would clog the spray equipment. The catalyst solution is added only as the paint is used and to the amount that can be applied in a 30 minute period.

NOTE: (1) The 3-week shelf life of the uncatalyzed S-13G paint shall not be exceeded except when the paint is stored under refrigeration (-10° F to +5° F). Under such conditions, the paint shall be considered usable until it jells or develops a stringy texture.

(2) The pot life of the catalyzed paint is approximately 30 minutes at moderate room temperature.

3.5 Application of S-13G Thermal Coating

3.5.1 Spray the paint on the primed aluminum surfaces to a thickness of 8 (-1 +2) mils using clean, dry, oil free Nitrogen as a pressurizing gas for the paint spraying. The pressure at the nozzle of the spray gun shall be determined by operator.

NOTE: A 5 micron air filter shall be used in the Nitrogen pressurizing line.

3.5.2 Allow the coated sample to air dry for 5 - 10 minutes between coats to permit solvent to evaporate.

3.5.3 The final coat shall be allowed to dry for a minimum of 16 hours before packaging. Six (6) to eight (8) coats are required to obtain the desired paint thickness.

3.5.4 Upon completion of the final drying cycle, cover the surface of the coated samples with Teflon FEP or Tedlar (or approved equal) film.

3. 5 Application of S-13G Thermal Coating (continued)

NOTE: The coating shall be sprayed in a uniform thickness, taking care not to cause sags and runs.

3. 5. 5 Lower plastic shroud over unit during curing cycle, but do not allow plastic shroud to come in contact with the thermal coated surface during cure cycle. Free circulation of dust-free air is necessary for proper curing.

NOTE: It is imperative that dust and debris be kept off the surface during the curing process.

3. 6 Storage of S-13G Thermal Coated Hardware

3. 6. 1 Parts coated with S-13G thermal coating shall be wrapped in clean, neutral, unplasticized film, (Tedlar, teflon FEP, Mylar, or equivalent) and then placed in a carrying case, nylon C film bag, or wrapped in paper and sealed with tape, as applicable.

3. 7 Transportation at Marshall

3. 7. 1 An unplasticized plastic shroud should cover the S-13G coated unit while being transported from one point to another at MSFC.

3. 8 Packaging for Shipment

3. 8. 1 The S-13-G thermal coated hardware should have a fitted, unplasticized polyethylene plastic film covering the painted surface.

3. 8. 2 All pressure points on the transporter should be protected with at least four layers of unplasticized polyethylene film in addition to Neoprene rubber pads at these points.

3. 9 Repair of S-13G Thermal Coating

3. 9. 1 S-13G painted surfaces that are merely soiled, but not damaged, are washed with DI water and wiped with ethyl alcohol. Painted surfaces that are damaged are sanded with 325 mesh carborundum paper, ethyl alcohol wiped and re-sprayed to obtain the desired thickness, using a small 3 oz. Paasch'e air brush (model AVT with an ARF 3 Nozzle). Primer application is accomplished using an ARFI Nozzle. When wet surfaces are damaged all of the paint is removed by wiping and washing with Toluene and the priming and painting process is repeated.

3.10 Recleaning

NOTE: Clean S-13G thermal coating only as directed. Never use an organic solvent.

3.10.1 At the latest possible time prior to launch, all S-13G thermal coated surfaces which are visibly contaminated shall be washed down with analconox detergent - demineralized water solution using a Nylon brush to remove fingerprints, oil, grease, dirt, dust and other contaminants collected on the surface.

3.10.2 Rinse the entire surface with copious amounts of demineralized or filtered tap water.

4. CONTROL REQUIREMENTS

4.1 Viscosity

Test using a No. 4 Ford Cup, 15 to 17 seconds.

4.2 Weight

The weight of one gallon of paint shall be 11.9 to 12.3 pounds.

MANUFACTURING PROCESS DATA FOR ZINC CHROMATE
PRIMING OF THE FUEL AND OXIDIZER CONTAINER BULKHEADS

1. SCOPE

1.1 This manufacturing process data covers the surface preparation and subsequent priming of the fuel and oxidizer container bulkheads with zinc chromate.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

● Nordson, hot-airless spray equipment with pole gun spray attachment.

● Fixture PP-370-7654

● Portable vacuum cleaner

2.2 Materials

● Masking tape (2 inch width)

● Alcohol - MIL-A-6091 A

● Lint-free wiping cloth

● Tack cloth

● Zinc chromate primer - MIL-P-8585

● Toluene - TT-T-548

● Xylene - TT-X-916

3. PROCEDURE

3.1 Priming

The fuel and oxidizer container bulkheads shall be primed with zinc chromate as follows.

- 3.1.1 Install work fixture PP-370-7654 on the Y-ring.
- 3.1.2 Inspect bulkhead surface for breaks in the conversion coating. Reclean and reconversion coat the abraded areas.
- 3.1.3 Mask off flange fitting areas, all weld seams, and all optical alignment decals on the bulkhead.
- 3.1.4 Mask off the area of the Y-ring below the clamps on the holding fixture.
- 3.1.5 Using ladder on work fixture PP-370-7654, wipe the entire surface of the bulkhead, fittings, and area above clamps on the Y-ring with a tack cloth.
- 3.1.6 Vacuum clean the crotch of the Y-ring using a portable vacuum cleaner. Use a clean lint-free cloth saturated with alcohol, and continue cleaning by wiping the crotch area until all visible contamination has been removed.
- 3.1.7 Wipe the entire surface of bulkhead, fittings, and area above clamps on the Y-ring with a clean, lint-free, cloth saturated with alcohol or Toluene. Dry the surface with a clean, lint free cloth.
- 3.1.8 Use the zinc chromate primer called out on the applicable engineering drawing, spray a film of primer on all un-masked areas that will be 0.0006 inch to 0.0008 inch thick when age cured.

NOTE: The wet-film thickness of the primer should gauge approximately .0012 inch to .0016 inch to produce the desired dry film thickness.

- 3.1.9 When the entire surface of the bulkhead, fittings, and the masked-off portion of the Y-ring have been primed to the desired film thickness, allow 30 minutes minimum for the film to set. Remove all masking tape from the bulkhead and Y-ring.

4. CONTROL

4.1 Personnel shall insure that the following controls are exercised as applicable.

4.1.1 Insure that all surfaces have been conversion coated before priming coat is applied.

4.1.2 Insure that clean solvents and wiping cloths are used.

4.1.3 Insure that the safety precautions outlined in National Fire Prevention Association Code 33, governing the painting of articles in an emergency painting area, are observed.

4.1.4 Insure that the zinc chromate primer is not sprayed at a temperature below 180 degrees Fahrenheit without making necessary adjustments to the hydraulic pressure. The normal hydraulic pressure is 600 pounds per square inch.

4.1.5 Insure that Toluene or Xylene is used to thin the zinc chromate primer in accordance with the manufacturer's instructions.

MANUFACTURING PROCESS DATA ZINC CHROMATE PRIMING OF S-IC COMPONENTS

1. SCOPE

1.1 This manufacturing process data covers the surface preparation and subsequent priming of the fuel and oxidizer container components with zinc chromate.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Nordson, hot-airless spray equipment with pole gun spray attachment
- Portable vacuum cleaner

2.2 Materials:

- Masking tape (2 inch width)
- Alcohol - MIL-A-6091A
- Lint-free wiping cloth
- Zinc chromate primer MIL-P-8585A
- Toluene - TT-T-548
- Xylene TT-X-916

3. PROCEDURE

3.1 Priming

The fuel and oxidizer container components are to be primed on all external surfaces only with zinc chromate.

3.1.1 Inspect gore and skin surface for breaks in the conversion coating. Reclean and reconversion coat the abraded and corroded areas.

3.1.1.1 Mechanically clean the areas using stainless steel wool, stainless steel wire brush, or aluminum oxide abrasive.

3.1.1.2 Wipe the surface with clean, lint-free cloths saturated with alcohol or Toluene.

3.1.1.3 Dry the surface with clean, dry, lint-free cloths.

3.1.1.4 Apply the conversion coating solution (Iridite 14-2 at 4 to 6 oz/gal) with a sponge. Contact time should be 1/2 to 1 1/2 minutes.

3.1.1.5 Rinse by repeated wiping with a sponge saturated with clean water.

NOTE: Thorough rinsing is essential to remove fluoride residue from the surface.

3.1.1.6 Remove excess water with a sponge and allow the surface to dry.

3.1.2 Wipe the entire metallic surface of the gore, skin, and Y-ring with a clean, lint-free cloth saturated with alcohol or Toluene. Be sure all residues from the taped areas have been removed. Dry the surface with a clean lint-free cloth.

3.1.3 Mask off flange fitting areas on all gores and 5 inches from all weld edges including the edges and reverse side on gores, skins and Y-ring. Seal internal side of the components (skin, gores, Y-ring) with a polyethylene bag by completely enclosing the internal side and taping the plastic on the external side 2 inches from the weld edge. Use maskant tape to seal off 3 inches more on the external side adjacent to and meeting the edge of the polyethylene bag.

3.1.4 Use the zinc chromate primer called out on applicable engineering drawing. Spray a film of primer on all unmasked areas (external surfaces only apply) that will be 0.0006 inch to 0.0008 inch thick when age cured.

NOTE: The wet-film thickness of the primer should gauge approximately 0.0012 inch to 0.0016 inch to produce the desired dry film thickness.

3.1.5 After the entire external surface of the gore, skin, and Y-ring has been primed to the desired film thickness, allow 30 minutes minimum for the film to set. Remove all the masking tape from the gore and cylinder.

4. CONTROL

4.1 Personnel are to insure that the following controls are exercised.

4.1.1 Insure that all surfaces have been conversion coated before priming coat is applied to the external surface.

4.1.2 Insure that clean solvents and wiping cloths are used.

4.1.3 Apply the zinc chromate primer to the T87 condition gore and skin soon after receiving inspection is completed to retard corrosion. All other parts in T37 condition that are artificially aged, will be primed after aging treatment.

4.1.4 Insure that the safety precautions outlined in the National Fire Prevention Association Code 33, governing the painting of articles in an emergency painting area, are observed.

4.1.5 Insure that the zinc chromate primer is not sprayed at a temperature below 180° F without making necessary adjustments to the hydraulic pressure. The normal hydraulic pressure is 600 pounds per square inch. square inch.

4.1.6 Insure that Toluene or Xylene is used to thin the zinc chromate primer in accordance with the manufacturer's instructions.

4.1.7 The zinc chromate priming operation will be accomplished once only. Any subsequent primer required will be made as a repair to abraded areas only.

APPLICATION OF S-13 THERMAL COATING

1. SCOPE

1.1 This manufacturing process data covers the surface preparation, priming and application of S-13 thermal coatings to the Instrument Unit and the Instrument Unit Adaptor.

2. EQUIPMENT AND MATERIALS2.1 Equipment

- Regular air-spray (Binks or equivalent)
- 1 pint cup
- Air pressure line regulated to 60 psi
- Gram balance
- Nylon brush
- Plastic shroud
- Wet film thickness tester (Nordson)
- Hobart mixer (or equivalent)
- Small paint brush

2.2 Materials

- SRC-05 Catalyst
- Special toluene
- Bulk paint

- General Electric Primer SS-4044
- Bulk paste
- Alconox detergent, or equivalent
- Demineralized water
- Wiping cloths
- Masking tape
- Methyl Ethyl Ketone
- Freon, Precision Cleaning Agent

3. PROCEDURE

3.1 Surface preparation

Prepare the surfaces for priming as follows.

- 3.1.1 Scrub all greasy surfaces with a solution of Alconox detergent and demineralized water utilizing a nylon brush.
- 3.1.2 Rinse the surface with copious amounts of demineralized water to insure removal of the detergent solution.
- 3.1.3 Wipe the surface with clean, lint free cloths to remove excess water from surface.
- 3.1.4 Lower a plastic shroud over unit during drying.
- 3.1.5 Allow surface to air dry for at least two hours before further processing.
- 3.1.6 Wipe with Freon Precision Cleaning Agent or MEK prior to priming the surface. Let dry.

3. 2 Priming: Prime surfaces as follows.

3. 2. 1 Use General Electric primer SS-4044 without dilution. Spray apply a thin film (.00025 to .00040 in.) over the entire surface to be painted.

3. 2. 2 Lower plastic shroud over unit during drying of primer.

3. 2. 3 Allow primer to air dry for one hour before further processing.

NOTES: The S-13 thermal coating will not adhere to an unprimed surface. Make certain that the entire unit is covered with primer.

3. 3 Mixing

Instructions for mixing the S-13 Thermal Coating are as follows.

3. 3. 1 Mix 2.1 grams of SRC-05 catalyst with 41.9 grams of special toluene.

3. 3. 2 Add mixture from paragraph 3. 3. 1 to 956.0 grams of bulk paint.

3. 3. 3 Use a metal spatula or paddle and stir for 10 minutes, or until the catalyst is thoroughly incorporated into the bulk paint.

NOTE: The pot life for S-13 thermal coating is approximately 2 hours @ room temperature. Do not mix more material than can be utilized within this period.

3. 4 Application

Apply the S-13 Thermal Coating as follows.

3. 4. 1 Apply a total of .009 inch thick coat of S-13 thermal coating by adjusting the spray pattern to put on .003 inch per pass for 3 passes.

3. 4. 2 Measure thickness with a wet-film thickness gauge (Nordson).

NOTE: S-13 thermal coating must not be applied in such a manner as to cause sags or runs.

3. 4. 3 Lower a plastic shroud over unit during curing cycle.

NOTE: Do not allow plastic shroud to come in contact with thermal coated surface during cure cycle.

3.4.4 Allow coating to cure, undisturbed, for at least 24 hours before further processing.

NOTE: It is imperative that dust and debris be kept off the surface during the curing process.

3.5 Storage of S-13 thermal coated hardware

Store all S-13 thermal coated hardware under a plastic shroud.

3.6 Transportation at Marshall

Cover the S-13 thermal coated instrument unit and adapter with an unplasticized plastic shroud while being transported from one point to another at MSFC.

3.7 Packaging for shipment

Package the S-13 thermal coated hardware as follows.

3.7.1 Cover the S-13 thermal coated hardware with a fitted, unplasticized polyethylene plastic film.

3.7.2 All pressure points on the transporter should be protected with at least four layers of unplasticized polyethylene film in addition to neoprene rubber pads at these points.

3.8 Repair of S-13 Thermal Coating

3.8.1 Mix 0.3 gram of SRC-05 Catalyst to 100 grams of bulk paste in a Hobart (or equivalent) mixing pan.

3.8.2 Allow the formula to remain in the mixer for at least 5 minutes.

3.8.3 Re-prime the area to be repaired with GE-SS-4044 primer with a small paint brush.

3.8.4 Allow primer to dry for at least 1 hour before paint is applied.

3. 8. 5 Apply S-13 thermal coating repair paste by trowelling (or with spatula), to the primed area.

NOTE: The S-13 coating paste material has a pot-life of approximately 1/2 hour.

4. CONTROL

4. 1 Recleaning

NOTE: Clean S-13 thermal control surfaces only as directed. Never use an organic solvent for cleaning.

4. 1. 1 At the latest possible time prior to launch, all S-13 thermal coated surfaces which are visibly contaminated shall be washed down with an Alco-nox detergent - demineralized water solution using a nylon brush to remove fingerprints, oil, grease, dirt, dust and other contaminants collected on the surface.

4. 1. 2 Rinse the entire surface with copious amounts of demineralized or filtered tap water to remove the detergent solution.

MANUFACTURING PROCESS DATA FOR APPLICATION OF S-13G THERMAL CONTROL COATING

1. SCOPE

1.1 This manufacturing process data covers the surface preparation, priming, application, and protection of S-13G thermal coatings to ATM hardware.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Spray booth, dry baffle or paint arrestor
- Spray gun, (Binks Model No. 18), equipped with pressure cup.
- Suction feed with fluid nozzle No. 66 and air cap No. 66S
- Pressure cap No. 80-210 or 80-212, fluid nozzle No. 63B, and air cap No. 63P.
- Strainer
- Viscosimeter, or No. 4 Ford Cup
- Electronic thickness tester
- Nylon gloves or expendable cotton gloves
- Five micron air filter
- Air brush, Paasch Model AU70, ARF-1 or ARF-3 Tip.
- Plastic shroud (Class 100 000 clean room or equivalent)

Materials

- SRC-05 Catalyst
- X99 thinner (toluene, U. S. P. , May be substituted)
- | <u>Ingredient</u> | <u>Parts by Volume</u> |
|-------------------|------------------------|
| Toluene | 40 |
| Xylene | 20 |
| Isopropanol | 20 |
| Butanol | 15 |
| Butyl Acetate | 5 |
- Bulk paint, S-13G
- General Electric Primer SS-4044
- Alconox detergent, or equivalent
- Demineralized water
- Wiping cloths
- Masking tape, pressure sensitive adhesive tape
- Number 60 Aloxite cloth (or approved equal)

3. PROCEDURE

3.1 Cleaning

Flush tank with water to remove the worst accumulation of dirt. Check the flow of water and make certain the drainage is working satisfactorily.

3.2 Paint Stripping

3.2.1 Work on a limited area at a time such as one gore section on the bulkhead and 4 foot wide section on the vertical portion of the tank.

Apply Cee Bee A-202 or Turco 5003 with a paint brush to make certain that the whole surface is covered completely. Allow sufficient contact time to loosen the paint.

3.2.2 Scrub the chemical solution on the surface to remove the paint and the primer.

3.2.3 After the primer is loosened, flush the surface with high pressure water to remove the loosened materials.

3.2.4 Repeat steps 3.2.1 to 3.2.4 if required, until all the paint and undercoat primer are removed from the tank. Orbital sanding may be required to remove primer from certain areas.

3.3 Oxide Removal

3.3.1 Wipe surface free of all oils with Methyl Ethyl Ketone solvent moistened cloth.

3.3.2 Mechanically clean tank by orbital sanding operation until all evidence of conversion coating is removed being very careful not to sand unevenly.

3.3.3 Wipe surface of tank clean with cloths moistened with MEK (Methyl Ethyl Ketone) solvent.

3.3.4 Continue all mechanical cleaning and solvent wiping operations until the conversion coat is removed from the surface of the metal.

4. CONTROLS

4.1 Safety

4.1.1 Check security of all scaffolding and personnel carriers such as high lifts, etc.

4.1.2 All personnel must be instructed to avoid physical contact with the chemicals. The corrosive nature of the chemicals will cause burns, therefore, protective clothing, splash proof goggles, gloves and boots must be worn. Avoid contact with rinse water.

4.1.3 The paint strippers and the MEK solution are flammable and all the fire hazard precautions must be taken. In addition, the fumes produced are irritating and ventilation must be adequate. Respirators or supplied air masks are recommended where access to fresh air is limited.

4.1.4 The tank should be protected with a plastic shroud after being cleaned because it will be out of doors and will need protection from the weather.

ZINC CHROMATE PRIMING OF ALL S-IC WELD AREAS

1. SCOPE

1.1 This manufacturing process data covers the surface preparation and subsequent priming of all external weld areas of the fuel and oxidizer containers.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Nordson, hot-airless spray equipment with pole gun spray attachment.
- Portable vacuum cleaner.

2.2 Materials

- Masking tape
- Alcohol MIL-A-6091A
- Lint-free wiping cloth
- Zinc chromate primer MIL-P-8585A
- Toluene TT-T-548
- Xylene TT-X-916

3. PROCEDURE

3.1 Priming

The external surfaces of all weld areas of the fuel and oxidizer container are to be primed with zinc chromate as follows.

3. 1. 1 The zinc chromate primer adjacent to weld areas is to be feather edged by hand methods.

3. 1. 2 Inspect all weld areas for breaks in the conversion coating. Re-clean and reconversion coat the abraded and corroded areas as follows.

3. 1. 2. 1 Mechanically clean using stainless steel wool, stainless steel wire bursh, or aluminum oxide abrasive.

3. 1. 2. 2 Wipe with clean, lint-free cloth saturated with alcohol or toluene.

3. 1. 2. 3 Dry the surface with clean, dry, lint-free cloth.

3. 1. 2. 4 Apply the conversion coating solution (Iridite 14-2 at 4 to 6 ox/gal) with a sponge. Contact time should be 1/2 to 1 1/2 minutes.

3. 1. 2. 5 Rinse by repeated wiping with a sponge saturated with clean water.

NOTE: Thorough rinsing is essential to remove fluoride residue from the surface.

3. 1. 2. 6 Remove excess water with a sponge and allow the surface to dry.

3. 1. 3 Wipe the entire external surface of all weld areas with a clean, lint-free cloth saturated with alcohol or toluene. Dry the surface with a clean, lint-free cloth.

3. 1. 4 Use the zinc chromate primer called out on the engineering drawing. Spray a film of primer on all weld areas and blend the primer into the primed areas to obtain an 0.0006 inch to 0.0008 inch thickness when age cured.

NOTE: The wet-film thickness of the primer should gauge approximately 0.0012 inch to 0.0016 inch to produce the desired dry film thickness.

3. 1. 5 When the entire surface of the weld area has been primed to the desired film thickness, allow 30 minutes minimum for the film to set.

4. CONTROL

4. 1 Personnel are to insure that the following controls are exercised as applicable.

- 4. 1. 1 Insure that all weld surfaces have been conversion coated and are free of corrosion before priming coat is applied.
- 4. 1. 2 Insure that clean solvents and wiping cloths are used.
- 4. 1. 3 Insure that the zinc chromate priming operation is to be completed immediately after the weld has been accepted by the cognizant laboratory.
- 4. 1. 4 Insure that the safety precautions outlined in National Fire Prevention Association Code 33, governing the painting of articles in an emergency painting area, are observed.
- 4. 1. 5 Insure that the zinc chromate primer is not sprayed at a temperature below 180°F without making necessary adjustments to the hydraulic pressure. The normal hydraulic pressure is 600 pounds per square inch. square inch.
- 4. 1. 6 Use only Toluene or Xylene to thin the zinc chromate primer in accordance with the manufacturer's instructions.

REMOVAL AND REPAIR OF ANODIC COATING
ON PARTS TO BE WELDED

1. SCOPE

1.1 This manufacturing process data applies to any aluminum that has been anodized prior to welding and where a subsequent protective finish is required to meet surface requirements. This process is directly applicable to the S-IC liquid oxygen system components.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Paint brushes
- Plastic buckets
- Aluminum wool
- Sponge

2.2 Materials

The following materials, or approved equal, shall be used.

- Cab-O-Sil
- Iridite 14-2 MIL-C-5541
- Tap water
- Ethyl alcohol - MIL-A-6091-A
- Trichloroethylene O-T-634
- Lead or plastic platers' tape
- Nitric acid - O-N-350

- Orthophosphoric acid - O-O-670
- Hydrofluosilicic acid
- Sulfuric acid - O-S-809
- Sodium dichromate - O-S-595
- Demineralized water
- Wiping cloths
- "Freon" PCA

2.3 Formulation

2.3.1 British Etch

The British etching solution, consisting of the etch solution and de-oxidizer, shall be formulated as follows:

2.3.1.1 Etch solution - Combine 120 grams (gms) Cab-O-Sil, 189 cubic centimeters (cc) nitric acid, 757 cc orthophosphoric acid, 113 cc hydrofluosilicic acid, 738 cc of water to make about 1/2 gallons of solution.

2.3.1.2 Deoxidizer solution - Combine 240 gms Cab-O-Sil, 294 cc of sulfuric acid, 189 gms of sodium dichromate, 1420 cc of water to make 1/2 gallon of solution.

2.3.2 Conversion coat solution - The conversion coat solution shall be formulated as follows.

2.3.2.1 Mix 4 to 8 ounces of Iridite 14-2 per gallon of water. The mixture shall have a pH in the range of 0.4 to 1.1.

2.3.2.2 Add 5 to 7 ounces of Cab-O-Sil per gallon of the solution.

2.3.2.3 Adjust pH of conversion coat solution with nitric acid when the pH rises above 1.1.

3. PROCEDURE

3. 1 Removal of coating - The coating shall be removed as follows.

3. 1. 1 Mask the part in such a manner that the area at a distance greater than 2 inches from the weld will not be etched. Either lead tape or plastic platers' tape shall be used.

3. 1. 2 Wipe the weld area with a clean cloth saturated with trichloroethylene. Allow the surface to dry.

3. 1. 3 Apply the ambient temperature etch solution by continuously and vigorously brushing for 5 to 8 minutes. Do not allow the etch solution to dry on the surface.

3. 1. 4 Rinse by sponging with tap or demineralized water until there is no visual indication of the etching solution.

3. 1. 5 Apply the ambient temperature deoxidizer solution by continuously and vigorously brushing for 2 to 5 minutes. Do not allow the deoxidizer solution to dry on the surface.

3. 1. 6 Rinse well by sponging with demineralized water until there is no visual indication of the deoxidizer solution.

3. 1. 7 Rinse the surface with alcohol and/or with "Freon" Precision Cleaning Agent and allow to dry.

3. 2 Repairs - After weld acceptance, all bare areas shall be repaired as follows.

3. 2. 1 Mechanically clean by hand or by machine grinding using non-electrolytic material (aluminum wool or aluminum oxide abrasive).

3. 2. 2 Wipe weld areas with a clean cloth saturated with trichloroethylene, or alcohol. Allow surface to dry.

3. 2. 3 Brush the conversion coat solution over the entire uncoated weld area.

3. 2. 4 Sponge or wipe off excess conversion coating solution from the weld area with demineralized water. Wipe all surrounding area with a dry cloth to remove all moisture. Allow surface to dry for a minimum of 24 hours at ambient temperature.

4. CONTROL

4. 1 Personnel shall insure that the following controls are exercised as applicable.

4. 1. 1 Prepare only enough of the etching and deoxidizing solutions for one use.

4. 1. 2 Use the solution as soon as practical after makeup.

4. 1. 3 If the solutions are not used on the day prepared, they must be kept tightly sealed. It may be necessary to replace evaporated water if the solution is not used for several days after formulation. Make-up water is added to the solution until the proper consistency is achieved.

PROCESS CONTROL AND OPERATING PROCEDURES FOR CADMIUM PLATING OF SPACE VEHICLE PARTS

1. SCOPE

1.1 This manufacturing process data covers the necessary precautions and procedures for process control of cadmium plating of space vehicle parts.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Handling fixtures
- Laboratory instruments (pH meter, titrating device, electroanalyzer, etc.)
- Sample beaker, 250 ml, minimum
- Pipette
- Erlenmeyer flask
- Magnetic stirrer
- Analytical balance
- Graduate cylinder (100 ml)
- Hull Cell

2.2 Materials

- Cadmium plating bath sample
- Sodium and Potassium cyanide
- Sodium carbonate

- Brightener (RHCO #20)
- Sodium hydroxide
- Distilled water
- Barium nitrate
- Sodium chromate
- La Motte Sulfo-orange, Phenolphthalein indicators
- Ethyl alcohol
- Potassium iodide
- Silver nitrate
- Nitric acid
- Platinum anode, copper gauge cathode
- Sodium chloride
- Litmus paper
- Cadmium anodes
- Sodium sulfide or equivalent soluble sulfide

3. PROCEDURE

3.1 Solution Control and Operating Conditions

3.1.1 Determine the total and free cyanide.

3.1.1.1 Pipette a 5.0 cc. sample into a 250.00 cc Erlenmeyer flask.

3.1.1.2 Add 100.0 cc of distilled water, 5.0 cc of 10 percent potassium iodide (KI) solution and 15.0 cc of ammonium hydroxide (NH_4OH).

3.1.1.3 Titrate with the standard 0.1 N silver nitrate solution until a faint yellow turbidity is produced. (The standard Ag NO₃ solution is prepared by weighing out exactly 17.0 grams of the chemically pure salt and dissolving to one [1] liter). The Total cyanide in oz/gal is equal to: 0.2626 x Ag NO₃ required for sample of solution. The sodium cyanide combined with the metallic cadmium is equal to:

1.74 x oz/gal metallic cadmium as determined in

$$\left[\frac{\text{ml} \times 0.2626 = \text{ml} \times \left(\frac{17\text{g}}{170 \text{ g/oz}} \right) (982 (0.134))}{5 \text{ ml.}} = \frac{\text{ml.} \times N \times 2 (\text{NACN}) (0.134)}{\text{ml. of sample}} \right]$$

The Free sodium cyanide (NACN) in oz/gal is equal to:

$$\text{Total sodium cyanide minus (1.74 x oz/gal Cd.)} \left[1.74 = \frac{4X \text{ weight of NACN}}{\text{M. wt of Cd.}} \right]$$

3.1.2 Determine carbonates.

3.1.2.1 Pipette a 10.0 cc sample into a 250.0 cc beaker.

3.1.2.2 Add 100.0 cc of distilled water and warm.

3.1.2.3 Add 10.0 percent barium nitrate solution while stirring, until no more precipitate forms. Each cc of the Ba (NO₃)₂ solution will precipitate an amount of carbonate equivalent to 1/2 oz/gal sodium carbonate. Allow to settle.

3.1.2.4 Filter, adding a few drops of Ba (NO₃)₂ solution to the first portion of filtrate to determine whether precipitation is complete. Wash with hot water.

3.1.2.5 Transfer the filter paper and precipitate to the beaker in which the precipitation was made.

3.1.2.6 Add 50 cc of hot distilled water and a few drops of 0.2 percent methyl orange solution.

3.1.2.7 Titrate with approximately 1.0 N HC1 until a permanent pink color is obtained.

Prepare the HC1 by diluting 83 cc of C.P. acid to 1 liter and standardizing as follows: A 5.0 cc sample of HC1 is pipetted into a 250.0 cc Erlenmeyer flask and 50.0 cc of distilled water is added. A 2 percent sodium hydroxide (NaOH) solution is added drop by drop until the solution is neutral to litmus. 1 cc of 2 percent Na₂ CrO₄ solution is added and the solution

is titrated with standard 0.1N silver nitrate until a red precipitate just forms. The normality of the HCl will be equal to:

$$\frac{\text{cc of 0.1N Ag NO}_3 \text{ required}}{50}$$

The sodium carbonate in oz/gal is equal to: $(0.706 \times \text{N of HCl}) \times \text{cc. HCl required for sample}$. The figures inside the parenthesis represent the factor for the standard HCl solution.

$$0.710 = \frac{\text{eq. wt of Na}_2\text{CO}_3}{10 \text{ ml of sample}} \quad (0.134)$$

3. 1. 3 Determine sodium hydroxide

3. 1. 3. 1 Pipette a 10.0 cc sample into a 250.0 cc Erlenmeyer flask.

3. 1. 3. 2 Add 10.0 cc of distilled water, 1 gram of solid sodium cyanide and 8 drops of La Motte Sulfo-Orange indicator.

3. 1. 3. 3 Titrate with standard 1.0 N HCl solution until the color changes from orange to yellow.

The sodium hydroxide in oz/gal equals: $0.536 \times \text{N of HCl} \times \text{cc of HCl required for sample}$. The figures inside the brackets represent the factor for the standard HCl solvent.

$$0.536 = \frac{\text{eq. wt of NOAH}}{10 \text{ ml of sample}} \quad (0.134)$$

3. 1. 4 Determination of Cd by Electrolysis

3. 1. 4. 1 Measure 2.0 mls. of the plating solution into a 250.0 mls. beaker. Under a fume hood, add 5 to 8 ml concentrate H_2SO_4 and 10 mls. HNO_3 , cool and dissolve in about 25 mls. distilled water. Add a few drops of phenolphthalein indicator and then add 20 percent NaOH to the phenolphthalein end point, in order to precipitate the Cd. as white cadmium hydroxide. Redissolve the precipitate with 10 percent KCN solution avoiding an excess. For any iron present to be dissolved, filter and dilute to about 220 to 230 mls volume. Electrolyze the solution, for 1 1/2 to 2 hrs. with agitation (magnetic stirrer or rotating anode) at 2 to 4 amperes; 4 to 6 volts using a platinum anode and a weighed copper gauge cathode. Spot check to determine if Cd. is plated out by adding a drop sample solution to one drop of 10 percent $\text{Na}_2\text{S.H}_2\text{O}$. If Cd is present--a yellow precipitation appears.

NOTE: The copper gauge cathode should be placed in the solution with the current on. If agitation is not possible, the current should be reduced. Remove the copper cathode with current on and rinse with ethyl alcohol and dry. Weigh the cathode to the nearest ten thousandths on analytical balance.

3.1.4.2 Calculate the cadmium.

$$\begin{aligned}\text{Weight of Cd.} \times 500 &= \text{g/L Cd.} \\ \text{g/L Cd.} \times 0.134 &= \text{oz/gal of Cd.}\end{aligned}$$

4. CONTROL

4.1 Control - The following controls and checks shall be made as noted.

4.1.1 Insure that beakers, pipettes and laboratory instruments are maintained in a clean condition.

4.1.2 Check the Cadmium metallic content, free and total cyanide, sodium hydroxide and carbonate content once per week.

4.1.3 Check normality of solutions against known standards once per week or as often as standards are made up.

PROCESS CONTROL AND OPERATING PROCEDURES FOR NICKEL
SULFAMATE PLATING OF SPACE VEHICLE PARTS

1. SCOPE

1.1 This manufacturing process data covers the necessary precautions and procedures for processing control of nickel sulfamate plating of space vehicle parts.

2. EQUIPMENT AND MATERIALS

2.1 Equipment

- Handling Fixtures
- Pipette
- Beaker (400 cc)
- Erlenmeyer flask

2.2 Materials

- Sodium pyrophosphate
- Ammonium chloride
- Potassium iodide
- Distilled water
- Ammonium hydroxide
- Sodium cyanide (0.5 N)
- Silver nitrate
- Glycerine

- Bromcresol purple indicator
- Sodium chromate
- Oxalic acid (C. P.)
- Nickel sulfamate bath sample

3. PROCEDURE

3.1 Solution Control and Operating Conditions

3.1.1 Pipette a 5 ml. sample into a 250 cc Erlenmeyer flask.

3.1.2 Add 75 cc of a solution made up by dissolving 50 grams of sodium pyrophosphate ($\text{Na}_4 \text{P}_2 \text{O}_7 \cdot 10 \text{H}_2\text{O}$), 50 grams of ammonium chloride and 2 grams of potassium iodide (KI) in water and diluting to 1 liter.

3.1.3 Add concentrated ammonium hydroxide ($\text{NH}_4 \text{OH}$) drop by drop until the solution shows a bluish color and smells faintly of ammonia. About three drops are necessary for a high pH solution.

3.1.4 Titrate with standard 0.5 N sodium cyanide (Na CN) solution until the precipitate which first appears just re-dissolves. Prepare the standard sodium cyanide (Na CN) solution by dissolving 25 grams of NaCN and 1.7 grams of Ag NO_3 in 1 liter of distilled water. Exactly 0.20 grams of pure metallic nickel are weighed out and dissolved in a 250 cc Erlenmeyer flask with a mixture of 5 cc. of concentrated sulfuric acid, 5 cc. of concentrated nitric acid, and 5 cc. of distilled water. The solution is evaporated until dense white fumes of sulfur trioxide (S O_3) are evolved. Cool the solution and add a slight excess of $\text{NH}_4 \text{OH}$ and boil. When the solution smells faintly of ammonia, cool to room temperature, add 75 cc of chloride pyrophosphate mixture and titrate with standard Na CN until the precipitate formed re-dissolves.

3.1.5 Calculate

The metallic nickel in oz/gal =

$$\frac{5.34}{\text{cc. of Na CN solution required for 0.20g Ni}} \times \text{cc. Na CN required for sampled solution}$$

3. 1. 6 Determine the chloride content.

3. 1. 6. 1 Pipette a 5 cc sample into a 250 cc Erlenmeyer flask.

3. 1. 6. 2 Add 50 cc of distilled water and 1 cc of 2 percent sodium chromate :
($\text{Na}_2 \text{CrO}_4$) solution.

3. 1. 6. 3 Titrate with standard 0.1 N silver nitrate solution. 1 cc of
0.1 N Ag NO_3 is equivalent to:

0.00535 gram ammonium chloride ($\text{NH}_4 \text{Cl}$)

0.00585 gram sodium chloride (Na Cl)

0.0110 gram nickel chloride ($\text{Ni Cl}_2 \cdot 6 \text{H}_2\text{O}$)

3. 1. 6. 4 Calculate.

The chloride expressed as $\text{NH}_4 \text{Cl}$ in oz/gal = $0.143 \times \text{cc}$ of
 Ag NO_3 used.

$$0.143 = \frac{0.1 \text{ N}}{5 \text{ ml}} \quad (\text{eq. wt. } \text{NH}_4 \text{Cl}) \quad (0.134)$$

The chloride expressed as NaCl in oz/gal =

$0.156 \times \text{cc}$ of Ag NO_3 used.

$$0.156 = \frac{0.1 \text{ N}}{5 \text{ ml}} \quad (\text{eq. wt. } \text{NH}_4 \text{Cl}) \quad (0.134)$$

The chloride expressed as NaCl in oz/gal =

$0.317 \times \text{cc}$ of Ag NO_3 used.

$$0.314 = \frac{0.1 \text{ N}}{5 \text{ ml}} \quad \frac{\text{wt. of } \text{Ni Cl}_2 \cdot 6 \text{H}_2\text{O}}{2} \quad (0.134)$$

3. 1. 7 Determine the Boric acid content.

3. 1. 7. 1 Pipette a 5 cc sample into an Erlenmeyer flask.

3. 1. 7. 2 Add 25 cc of C. P. glycerine $\text{C}_3 \text{H}_5 (\text{OH})_3$ and 1/2 cc. of Brom-
cresol purple indicator.

3.1.7.3 Titrate with standard 0.1 N Na OH solution until the color changes to dark green and then to purple. The Standard 0.1 N NaOH is prepared by dissolving 4 grams of NaOH in distilled water and diluted to 1 liter. Exactly 0.20 grams of C. P. oxalic acid ($\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) is weighed out and dissolved in 100 cc. of distilled water. A few drops of Bromcresol purple are added and the solution is titrated with NaOH solution until the solution turns blue.

One cc. of 1.0 N is equivalent to 0.0619 grams of boric acid so that the boric acid in oz/ gal =

$$\frac{5.25}{\text{cc. of NaOH required for .20 grams oxalic acid}} \times \text{cc. of NaOH used.}$$

The figure inside the brackets represent the factor for the standard NaOH solution.

$$5.25 = \frac{(\text{wt. of oxalic acid})}{7.5} \frac{\text{eq. wt. of H}_3\text{BO}_3}{\text{eq. wt. of H}_3\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O} (200)}$$

4. CONTROL

4.1 Control - The following controls and checks shall be made.

4.1.1 Determine the nickel content by titration, chloride and boric acid content once per week.

4.1.2 Insure that beakers, pipettes and all laboratory instruments are maintained in a clean condition.

4.1.3 Check the normality of solutions against known standards once per week or as often as solutions are made up.

MANUFACTURING SPECIFICATION FOR THE PAINTING
OF SPACE LAUNCH VEHICLE SURFACES

1. SCOPE

1.1 Scope

This manufacturing specification designates the Process Engineering Laboratory requirements for finishing exterior surfaces of space launch vehicles.

1.2 Applicability

The requirements set forth in this specification shall be applicable for the following finishes, as applied to the base metals listed in paragraph 1.2.2.

1.2.1 Finishes

- Type I Phosphate metal conditioner
- Type II Wash primer - Pretreatment coating
- Type III Conversion and anodic coatings for aluminum
- Type IV Zinc chromate for aluminum or steels
- Type V Quick drying enamels, applied over primers

1.2.2 Base Metal

Finishes covered in this specification are for use on the following metals.

1.2.2.1 Steels

- Alloy steels, Grades - 4130, 4140, 4340, and 8630
- Stainless steel, Grades - 301, 304, 316, 321, and 347

- Martensitic stainless, Grade -410
- Precipitation hardening, stainless, Grade 17-7PH

1. 2. 2. 2 Aluminum alloys

The finishes specified for aluminum shall be applicable to aluminum alloys - 2014, 2024, Bare; 2024 Clad; 5052; 5456; 6061; and 7075.

2. APPLICABLE DOCUMENTS

2. 1 Governmental

The following documents, of the issue in effect on the date of use of this specification, form a part of this specification to the extent indicated herein.

SPECIFICATIONS

Federal

Q-A-51C	Acetone
Q-A-396	Alcohol, Ethyl
Q-T-634a	Trichloroethylene, Technical
QQ-P-416a	Plating, cadmium (Electrodeposited)
TT-C-595	Color
TT-N-97 (2)	Naptha, Petroleum, Aromatic
TT-T-266A	Thinner; Dope and Lacquer
TT-T-548b	Toluene (for use in Organic Coatings)
TT-X-916	Xylene (for use in Organic Coatings)

Military

MIL-C-490A Cleaning and preparation of Ferrous and
Zinc Coated Surfaces

MIL-S-5002 Surface Treatments

MIL-C-5541 Chemical Films for Aluminum and
Aluminum Alloys

MIL-E-5556A Enamel, Camouflage, Quick Drying

MIL-P-6808A Primer Coating, Zinc Chromate, Application

MIL-C-8507B Coating, Wash Primer, Application

MIL-P-8585A Primer, Zinc Chromate, Low-Moisture
Sensitivity

MIL-A-8625 Anodic Coatings for Aluminum Alloys

MIL-I-10428A Isopropyl Alcohol, Technical

MIL-C-10578 Metal Conditioner Compound

MIL-C-15328A Coating, Pretreatment (Formula No. 117
for Metals)

MIL-S-15847A Spray Guns

MIL-F-18264B Finishes, Organic

DRAWINGS

George C. Marshall Space Flight Center

10410239 Painting Pattern for Saturn Vehicle

STANDARDS

Federal

FED Test Method
STD-141

Paint, Varnish, Lacquer, and Related
Methods of Inspection, Sampling and
Testing

FED-STD-595

Colors

(Copies of specifications, standards, drawings and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Other Publications

- "Airless Spray Coating Systems" - Instruction Manual.

Copies may be obtained from Nordson Corporation, 1000 Franklin Ave., Amherst, Ohio.

- "Paint Stripper" - Bulletin. (No. 4260) Copies may be obtained from Turco Products Co., 6135 S Central Ave., Los Angeles 1, California.

- Iridite #14-2 Al-Coat Bulletin. Copies may be obtained from Allied Research Products, Inc., 4004-06 East Monument Street, Baltimore 5, Maryland.

- Deoxidine #624 Bulletin. Copies may be obtained from Amchem Products, Inc., Ambler, Pennsylvania.

- Wash Primer -Bulletin (S-2201-M). Copies may be obtained from The Stoner-Mudge Co., 2000 Westhall, Pittsburgh 33, Pennsylvania.

3. REQUIREMENTS

3.1 General

No deviation from the requirements of this manufacturing specification shall be permitted without prior written approval of the MSFC initiating activity.

3.2 Material

Unless otherwise specified, all materials used in finishing and coating the space vehicle and its component parts shall conform to the requirements of applicable specifications. The addition to the paint or primers of any material other than prescribed thinners is prohibited.

3.2.1 Surface treatment materials

3.2.1.1 Metal conditioner and rust remover for steels

A phosphoric acid compound conforming to Specification MIL-C-10578, Type I (Wash-off) shall be used to prepare the steel surface and to improve paint adhesion.

3.2.1.2 Deoxidine (or equal)

A material for the removal of the oxide film from aluminum and aluminum alloys.

3.2.1.3 Conversion coatings and anodic film

Aluminum metal conditioners conforming to Specification MIL-A-8625 or Specification MIL-C-5541 using sulphuric or chromic acid as required, shall be used for the production of these coatings or films.

3.2.1.4 Cadmium plate

Electroplate, as required by engineering drawing, and process in accordance with Specification QQ-P-416a.

3.2.2 Primers

3.2.2.1 Coating, pretreatment

The primer coating used on steel parts shall conform to Specification MIL-C-15328A (contains 2 percent phosphoric acid) as furnished by the Stoner-Mudge Co., or the Sherwin Williams Co. P60G1 formulation, or equivalent. It is not intended as a protective coating in itself.

3. 2. 2. 2 Zinc chromate, low moisture sensitivity

Primer for use on aluminum and steel parts shall conform to Specification MIL-P-8585A, as furnished by Sherwin-Williams Co. or Brown Manufacturing Co.

3. 2. 3 Enamels

3. 2. 3. 1 Camouflage, quick drying

The enamels used as final coats on the exterior surfaces of the space vehicle and for lettering shall conform to Specification MIL-E-5556A with color designations as specified on the applicable engineering drawings.

3. 2. 3. 2 Heat resistant coatings

High temperature paint for use on the target areas for CZR camera tracking is furnished by the Engineering Materials Branch of Marshall Space Flight Center (M-S & M-M) and designated (O) R-1-16-17 white and CB-1-15-10 black.

3. 2. 4 Thinners

3. 2. 4. 1 Alcohol

The alcohol used in the acid-diluent of the pretreatment coating, Specification MIL-C-15328, shall be ethyl alcohol conforming to Specification O-A-396. Isopropyl alcohol may be substituted for ethyl alcohol on an equal volume basis, if it meets Specification MIL-I-10428A.

3. 2. 4. 2 Toluene - xylene

The thinner used for dilution of the zinc-chromate primer coating, Specification MIL-P-85A shall be toluene conforming to Specification TT-T-548. To obtain slower drying a portion of the toluene may be replaced by Xylene conforming to Specification TT-X-916.

3. 2. 4. 3 Naphtha

The thinner used for diluting enamels conforming to Specification MIL-E-5556A shall be aromatic, petroleum naphtha conforming to Specification TT-N-97, Type I, Grade B.

3. 2. 5 Cleaning solvents

3. 2. 5. 1 Acetone

The solvent, used for removing oil, grease, wax, markings and other solvent soluble contaminants from metal surfaces prior to subsequent treatments, shall conform to Specification O-A-51c.

3. 2. 5. 2 Naphtha

The solvent, used for removal of the last traces of contaminants just prior to applying synthetic enamels, shall be aromatic naphtha conforming to the Specification TT-T-266a.

3. 2. 5. 3 Trichloroethylene

Trichloroethylene conforming to Specification O-T-634a may be required to remove contaminants or strains difficult to remove by other solvents.

3. 2. 6 Paint stripper

A product that is capable of removing the complete coating system without deleterious effect on the metal, e. g. - Turco Paint Stripper 4260BX or equal. It shall remove the coating system within 30 to 45 minutes without undue scrubbing. It shall be a product that will wash off with a water rinse, leaving no residue.

3. 3 Equipment

3. 3. 1 General

The equipment used in conditioning and applying surface finishes on the Saturn space launch vehicle shall be capable of obtaining a finish that will comply with the requirements of this Specification, as set forth in subsequent sections.

3. 3. 2 Spray equipment

The spray gun and accessories shall be of such type as to adequately produce a satisfactory finish. It shall conform to Specification MIL-S-15847 or be of the airless, spray-coating type as supplied by the Nordson

Corporation, Amherst, Ohio, or equal. The airless equipment, when it is used, shall consist of a pumping system, high-pressure, paint heater, and an airless, spray gun with applicable spray nozzles and accessories for each service. Full details for use of the airless equipment are included in the instruction manual of the Nordson Corporation.

3.3.3 Heat lamps

Infra-red heat lamps of ample number and size to effect uniform distribution of heat for drying or baking of parts shall be used. They shall also be used where localized heating is necessary. Heat lamps shall be protected from breakage, by falling objects, by protecting them with wire mesh.

3.3.4 Cradles and holders

Suitable cradles, holders, and hoists shall be provided to accomplish the efficient handling of components. This includes the ability to rotate the fuel and LOX tanks and to provide proper display and easy access for spray painting of other components.

3.3.5 Crane

An overhead crane of the rail type with sufficient capacity to handle any vehicle components to be painted shall be available.

3.3.6 Cloths, tack-rags, disposable fiber or paper wiping materials

Only new, contaminant-free, lint-free, desized, mill-end cloths or material specified below shall be employed in these paint finishing procedures. Laundered shop cloths are permitted only when the requirements of paragraph 6.2 of Specification MIL-F-18264 are met. Disposable fiber or chemically treated paper wiping material may be used in prepainting procedures provided there is no alkalinity, silicone, water or organic solvent leachable sizing or residues present.

3.4 Facilities

3.4.1 Spray room

Paint finishing shall be conducted in an area adequately ventilated by means of a positive pressure air system effecting at least three changes of

air per hour. All air shall be admitted through filters which will exclude dust and dirt. The air shall be introduced in a manner to minimize turbulence and excessive air currents which might damage the paint film during painting and drying operations. All doors and windows shall be closed during the painting and drying cycles. This area shall be utilized for cleaning and conditioning components which are too large for the usual dip or immersion equipment. It shall also be used for pretreating, priming, finish coats, air-drying, and infra-red baking of components too bulky for the regular baking oven. The room shall be equipped with all the necessary equipment to effect completely satisfactory finishes, and it shall possess adequate illumination for the work to be done.

3. 4. 2 Exhaust system

To minimize explosion hazards and to collect excess paint and fumes that would be exhausted to the atmosphere, the spray room shall be equipped with a water curtain of adequate size.

3. 4. 3 Spray floor

The paint spray area shall be equipped with a gridded, metal flooring, over flowing water, or the floor shall be constructed of skid resistant material which will drain completely after scrubbing.

3. 4. 4 Spray room - cleanliness

The spray area shall be maintained in a clean and orderly condition. The walls and floor shall be so constructed that they can be scraped clean with spark resistant tools. All refuse, rags and other extraneous materials shall be kept in covered containers. The area shall be thoroughly cleaned just prior to each painting operation.

3. 5 General Procedure

3. 5. 1 Cleaning - general

All metal surfaces shall be thoroughly cleaned and dried prior to the application of surface treatments, such as conversion coating or wash primer. Meticulous cleaning of space vehicle components cannot be over emphasized, since this factor is of prime importance in obtaining a satisfactory paint

finish. Cleaning shall be accomplished with solvents, detergents and processes which have no deleterious effect on the surface and which produce surfaces satisfactory for receiving subsequent finishes.

NOTE: Reclaimed thinners and solvents shall not be used for cleaning, as these materials may leave a grease film which will cause poor adhesion of the next coat.

3. 5. 2 Steel cleaning

All steel surfaces shall be cleaned and pretreated prior painting. The cleaning treatment shall leave the metal surface bare and free of oil, grease, dirt, scale, rust or other foreign matter such as marking ink.

3. 5. 2. 1 Acetone swabbing

Regardless of previous treatment, steel parts shall be hand swabbed with clean cloths soaked in acetone (or equivalent) dispensed from plunger type safety cans.

3. 5. 2. 2 Synthetic thinner wiping

Acetone swabbing shall be followed by hand wiping with lint-free cloths meeting paragraph 3. 3. 6 of this specification, dampened with synthetic enamel thinner which meets paragraph 3. 2. 5. 2 of this specification. This cleaning operation shall be done just prior to the application of the coating.

3. 5. 3 Aluminum cleaning

Surfaces of aluminum shall be cleaned, deoxidized and pretreated prior to painting.

3. 5. 3. 1 Cleaning

The cleaning treatment shall leave the metal surface bare and free of oil, grease, dirt, oxides or other foreign matter such as marking ink.

NOTE: The surface shall be sufficiently free of contaminants for the deoxidizer to leave a properly etched surface.

3. 5. 3. 2 Deoxidizing

The aluminum components to be processed in the paint shop shall be treated with Deoxidine (or equal) to remove the oxidized surface coating. The surfaces shall be treated in sections by hand brushing the deoxidizer onto the accessible areas (tank surfaces) and by spraying those areas of lesser accessibility (bulkheads). The sections shall be no larger than can be treated and rinsed in 3 to 5 minutes. The deoxidizer shall be removed by thorough water washing. The entire surface to be primed shall be kept wet with water until it is treated with the conversion coating material.

3. 5. 4 Preparation of surfaces

3. 5. 4. 1 General

The particular surface treatment and preparation of components shall be specified in engineering orders or drawings.

3. 5. 4. 2 Steel phosphate treatment

Steel components as specified by engineering orders shall be conditioned with MIL-C-10578, Type I (wash off), Metal Conditioner and Rust Remover (phosphoric acid base) as specified by MIL-C-490A and MIL-STD-171.

NOTE: Spray equipment shall preferably be used to apply the metal conditioner.

A. Clean surfaces - The conditioner shall wet the surface only long enough to just slightly etch the metal.

B. Rusted surfaces - The conditioner shall be applied in sufficient quantity and left in contact with metal for 2 to 10 minutes to loosen any rust present.

C. Rinse - Rinse the surface with clean water (preferable hot water) at 160° F and allow to dry thoroughly.

NOTE: Zinc chromate primer should be applied as soon as possible to prevent surfaces from reoxidizing or corroding.

3.5.4.3 Aluminum conversion coating

Aluminum surfaces specified by engineering orders to be treated with a conversion coating material shall conform to paragraph 3.2.1.3 of this specification. It shall be applied immediately after the deoxidizer. The material shall be applied with spray equipment or by hand brushing, as applicable, and in accordance with the requirements of Specifications MIL-S-5002 and MIL-C-5541, and with Iridite #14-2 (or equal) bulletin.

3.5.5 Application of primers

3.5.5.1 General

The paint type protective coatings (primers) shall be applied by spraying in such a manner as to ensure a smooth, continuous film that is free of imperfections, such as dried overspray, runs, sags, blisters or orange peel. Any specks, occlusions, or roughness of primers will carry through to the topcoat and impart undesirable roughness to the final finish, therefore extreme care shall be exercised in the application of the primers.

3.5.5.2 Steel pretreatment coating (Formula No. 117)

Stainless steel components and those parts so designated by engineering orders shall be spray coated with one or two coats, as required, of Wash Primer, Specification MIL-C-15328A as furnished by the Stoner-Mudge Co., or Sherwin Williams P60G1, or equivalent, prior to application of subsequent coats. It is used to increase the adhesion of the coating system. A second coat may be applied within 15 to 30 minutes after application of the first coat.

NOTE: Parts shall be coated with zinc chromate primer or enamel as soon as practical (preferable within 24 hours), since the wash primer loses its effectiveness after a short exposure to adverse weather conditions.

NOTE: The pretreatment is most effective when freshly mixed and must be used within 8 hours after the addition of the acid component. The wash primer shall be applied in conformance with Specification MIL-C-8507B.

3.5.5.3 Aluminum priming

Aluminum components shall be primed per paragraph 3.6.

3.6 Zinc Chromate Primer, Low Moisture Sensitivity

3.6.1 Base metals

This primer is suitable for application to steels or aluminum whose surfaces have been treated for paint adhesion. This primer may be used with or without a top coating.

3.6.2 Inspection

Prior to priming, all previously treated surfaces shall be visually examined for cleanliness and for the satisfactory condition of all pretreated surfaces.

3.6.3 Primer

Zinc chromate primer meeting the requirements of Specification MIL-P-8585A shall be sprayed or brushed onto parts in accordance with the requirements of Specification MIL-P-6808A. When sprayed, the coating shall be free of runs, sags, orange-peel, blisters or roughness that will carry through to a subsequent coat. On single applications, the dry-film thickness shall be 0.3 to 0.4 mil. Specification MIL-P-6808A shall govern as to methods of use, thickness, drying, baking and recoating time.

NOTE: If parts received a pretreatment wash primer, prior to zinc chromate priming, they shall dry for a minimum of 6 hours before proceeding with top coats.

NOTE: Zinc chromate primer shall be used on all surfaces except those exempted by engineering orders.

3.6.4 Sanding

When second primer coats are required, the first primer coat shall be air dried for a minimum of 1 hour prior to scuff sanding with #400 sandpaper. To obtain the required smoothness, sanding shall be permitted only to remove small specks that might carry through to the topcoats.

3.6.5 Tack rag

Whether primer is sanded or not, a wipe down with a tack rag is required immediately prior to top coating, if any undue delay is encountered which might cause contamination.

3.7 Quick Drying Enamels

3.7.1 Inspection

Prior to the application of top coats, all primed components shall be inspected for satisfactory workmanship and quality of the primer coat.

3.7.2 Legend

The following enamels meeting the requirements of Federal Standard 595 shall be employed on the Saturn space launch vehicle.

- #31136 Red, Lusterless
- #33538 Orange-yellow, Lusterless
- #37038 Black, Lusterless
- #37875 White, Lusterless

The color, red, shall be used for lettering "United States"; the color orange-yellow, for the legends; the pigment, black, for all other markings; and the color, white, for the remainder of the vehicle requiring an enamel topcoat. Any deviations shall be specified by the engineering drawing.

3.7.3 Application

The enamel shall be applied by spraying, using the airless spray process and equipment, or equal, to those areas of large dimensions. The white enamel shall be applied first and the lettering subsequently. The lettering shall be applied by hand brushing or by masking-off and spraying. The white and lettered areas are to be masked off prior to application of the black enamel.

Unless enamel is specified on the drawings, all surfaces, required to be primed with zinc chromate, shall be left in the as-primed state.

Specification MIL-F-18264B shall govern the methods of application and the handling of enameled parts.

Dry film thickness for enamels shall be a maximum of 1 ml.

3.7.4 Drying time

The first coat of enamel shall have dried for a minimum of 45 minutes before application of a second coat. The second coat, if required, shall be applied within a maximum of 96 hours. If the second coat is delayed overnight or longer, the surface shall be wiped down with solvent to assure adequate adhesion. The final coat shall be allowed to dry a minimum of 16 hours before removal from the painting area.

3.7.5 High temperature paint

CZR camera tracking target areas shall be coated with high temperature paint, covered in paragraph 3.2.3.2 of this specification. The areas around the continuous lights shall be painted directly on the bare metal with (O)R-1-16-17 White, High Temperature and CB-1-15-10 Black, High Temperature. Method of surface treatment, application and coating thickness shall be as shown on Drawing 10410239, Note #9.

3.8 Miscellaneous Requirements

3.8.1 Surface refinishing or touch-up

Areas on the completely assembled vehicle, where the finish has been damaged or inadequately applied, shall be repaired as follows.

3.8.1.1 Scratches, abrasions and thin areas

If bare metal has been exposed, the edges adjacent to the bare area shall be tapered by wet sanding with wet-dry sand paper. Any area being repaired shall be tack-ragged to remove excess dust. It shall be swabbed with a water moistened cloth and then wiped clean with a cloth dampened by an approved solvent that will not damage the adjacent paint.

A. Aluminum components - Aluminum components shall be manually brush treated with Iridite 14-2 or equal.

B. Steels components - Steel components shall be treated with the primer applicable to the type of steel.

C. Sanding - A small area of the old finish surrounding the damaged region thus treated shall be sandpaper feathered and wiped clean prior to priming.

D. Re-prime - Zinc chromate primer conforming to Specification MIL-P-8585A shall be applied by hand to the damaged area.

E. Top coat repair - Areas requiring enamel shall, preferably, be sprayed, otherwise, it shall be applied by hand brushing. Areas not requiring enamel shall be left in the primed condition.

3.8.2 Stripping

Finishes that have been poorly applied, damaged by handling, or by static firing, over an area too large for touch-up, shall be stripped to the bare metal with an appropriate paint stripper, such as Turco No. 4260 or equal. The surfaces shall be refinished per the appropriate sections (3.5, 3.6, and 3.7) in this specification.

3.8.3 Masking

One square foot of area around the fin alignment markings on all outboard fuel and LOX containers and all cradle and erection target areas shall be masked off before spraying with enamel. Masking or covering shall be applied to electrical plugs, water quench connectors, threaded or screw parts, turbine exhaust duct, drain lines, and insulating parts of all antennas. These parts shall not be coated nor shall spray directed on adjacent parts or areas fall upon them. Areas around the target lights shall be masked as soon as the bare metal is cleaned.

3.8.4 Tapes

Tapes used in masking applications shall not impair the properties of, or discolor, the paint film. Tapes shall be removed as soon as practical; however, to avoid staining, they should not remain longer than 2 hours.

4. QUALITY ASSURANCE PROVISIONS

4.1 Cleanliness

Cleanliness of cleaned, etched surfaces shall be checked by employing the water break test outlined in paragraph 5.1.4.2.1 of Specification MIL-F-18264B.

4. 2 Inspections

Inspections shall be made at various stages of cleaning, surface treating, priming, and painting to ascertain that this specification is followed. The inspections are to be made by the metals-finishing supervisor and by Quality Division representatives as required.

4. 2. 1 Tests

Prior to use, coating materials shall be examined for compliance with the individual materials, specifications, and the manufacturer's recommendations.

No mixing of different manufacturer's paints shall be permitted, though they meet the requirements of the same specification. No interchange of thinners or diluents shall be permitted. Paint from one manufacturer shall be used to paint the entire surface of the vehicle or a component. This limitation shall apply to the whole coating system.

No zinc chromate primer shall be used which is more than 1 year old from the date of manufacture.

4. 2. 2 Paint sequence break

When painting sequence is interrupted over night, a solvent cleaning of the surface is required. A final wipe-down shall also be performed as required immediately before painting to insure a proper paintable surface.

4. 3 Paint Film Thickness

The dry film thickness of coatings applied under this specification shall be as noted.

- | | |
|---|-----------------------|
| ● Wash primer | 0.0002 - 0.0003 inch |
| ● Zinc Chromate primer | 0.0003 - 0.0004 inch. |
| ● Zinc Chromate primer plus
one coat of enamel | 0.0008 - 0.0010 inch. |
| ● Zinc Chromate primer, 2 coats | 0.0007 inch. |

- Wash primer plus Zinc Chromate 0.0017 - 0.0022 inch.
primer plus 2 coats of enamel
- Insignias, add 0.001 inch.

4.4 Smoothness and Uniformity

Specks and bumps, as indicated by dragging the finger tips across the surface, shall be removed by careful sanding. No overspray, seediness or roughness shall be permitted. Coatings shall also be visually checked for color, highing power and appearance.

4.5 Adhesion

The final painted surfaces shall be inspected for paint adhesion by tests set forth in Specification MIL-C-490A, paragraph 4.2.4.2 or MIL-F-18264B, paragraph 8.5.

5. PREPARATION FOR DELIVERY

5.1 Preservation and Packaging

Finished, painted components shall be handled as called out by engineering drawings or by the terms of contract.

5.2 Marking

Components shall be identified as engineering drawings or as contract dictates.

6. NOTES

6.1 Intended Use

This manufacturing specification, developed by the Process Engineering Laboratory of the George C. Marshall Space Flight Center, is intended for use in cleaning, preparation, priming and painting of surfaces of space launch vehicles.

6.2 Safety Precautions

6. 2. 1 Personnel safety

All necessary safety precautions regarding toxicity and industrial health hazards shall be taken by painting personnel under supervision of paint foreman and safety engineer in accordance with MSFC instructions and regulations. Adequate paint-spray respirators, non-sparking shoes and protective clothing shall be available.

6. 2. 2 Electrical grounding

Safety precautions regarding storage of finishing materials and thinners, non-sparking floors, handling equipment, vapor and explosion proof lights, fire prevention apparatus as contained in Specification MIL-C-18187 shall be applicable and observed in painting of space launch vehicles.

Prior to painting, the vehicle or components shall be grounded to prevent explosions caused by discharges of static electricity.

6. 2. 3 Paint storage

Paint finishing materials shall be stored in rooms with fire proof racks. The temperature shall not be lower than 50°F nor higher than 80°F. Materials subjected to 100 degrees Fahrenheit on more than three occasions, must pass qualification tests to affirm a lack of deterioration in quality.

NOTICE: When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian

Process Engineering Laboratory
George C. Marshall Space Flight Center

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING SPECIFICATION FOR SILK-SCREEN REPRODUCTIONS

1. SCOPE

1.1 Scope

This specification covers the approved Process Engineering Laboratory manufacturing requirements for methods of duplication by means of the silk screen process.

1.2 Classification

The types of silk-screen stencils covered by this specification are as listed.

- Block-out stencils
- Resist stencils
- Paper stencils
- Shellac or Lacquer film stencils
- Cellophane or Celluloid stencils
- Photo - stencils

1.2.1 Screening

The generic term, silk-screen process, shall cover both natural silk and fine mesh, metallic screens, which may serve as an integral screen and stencil or which may serve as a base to which the stencils adhere.

1.3 Applicability

These methods are applicable to all printable materials processing reasonably smooth surfaces.

1.4 Limitations

Only the fineness of detail and the inability to produce shading limits the usefulness of each type of silk screen stencil.

2. APPLICABLE DOCUMENTS

2.1 Government

The following documents, of the issue in effect on the date of use of this specification, form a part of this specification to the extent indicated herein.

SPECIFICATIONS

Federal

O-P-559	Potassium Bichromate, Technical Grade
O-S-603B	Sodium Hydroxide, Caustic Soda, Photographic
TT-I-558b	Ink, Marking Stencil, Opaque, for Nonporous Surfaces
TT-I-559b	Ink, Marking Stencil, Opaque, for Porous Surfaces
TT-L-26	Lacquer, Cellulose Nitrate, Brushing, Gloss
TT-P-98 (1)	Paint, Stencil Flat
TT-S-271B	Shellac, Dry, Orange and other Lacs

Military

JAN-E-199 (2)	Ether Diethyl
JAN-G-338	Glue, Animal
JAN-A-489	Acetone, Technical
MIL-E-7729A	Enamel, Gloss for Aircraft Application

MIL-A-10165

Ammonium Hydroxide

MIL-E-1-242

Enamel, Stencil, Gloss

MIL-L-52043 (ORD)

Lacquer, Semi-Gloss, Cellulose
Acetate

(Copies of specifications, standards, drawings, and publications required by contractor in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Other Publications

The following documents form a part of this specification. Unless otherwise indicated, the issue in effect on the date of issuance of this specification shall apply.

E. I. DuPont DeNemours and Company, Inc.

Bulletin V8-457

"Elvanol", Polyvinyl Alcohol, and
"Elvacet", Polyvinyl Acetate, for
Coating Stencil Screens.

(Copies of this publication listed above may be obtained from E. I. DuPont DeNemours and Company (Inc.), Wilmington 98, Delaware.)

Eastman Kodak Company

Kodak Pamphlet No. Q-24 Industrial Uses of Kodak Photo Resist

Kodak Pamphlet No. Q-15 Kodak Ektagraph Film

(Copies of these publications listed above may be obtained from Eastman Kodak Company, Rochester 4, New York,)

The Naz-Dar Company

Catalog No. 30

Catalog of Screen Process Colors,
Equipment, Supplies

(Copies of this publication listed above may be obtained from The Naz-Dar Company, 461 Milwaukee Avenue, Chicago 10, Illinois.)

3. REQUIREMENTS

3.1 General

No deviation from the requirements of this manufacturing specification shall be permitted without prior written approval of the MSFC initiating activity.

3.2 Materials

3.2.1 Silk

Bolting cloth of the desired width and of single-X or double-X grade shall be used in the following meshes as required by the reproduction.

<u>No.</u>	<u>Mesh Count</u>	<u>No.</u>	<u>Mesh Count</u>
1	48	11	116
2	54	12	124
3	58	13	130
4	62	14	139
5	66	15	148
6	74	16	157
7	82	18	170
8	86	20	180
9	97	25	200
10	109		

3.2.1.1 Silk alternates

Dacron and nylon have proven to be acceptable substitutes for silk, even for fine detail.

3.2.2 Metallic screening

Stainless steel, brass, copper, or phosphor-bronze wire in meshes from 80 to 250 may be used when the service demands durability, stability and accurate reproductions.

3.2.3 Printing base

While the silk-screen printing process is adaptable to almost any material, its usefulness can be maintained by printing only grease-free, stain-free and dirt-free items.

3.3 Stencils

3.3.1 Block-out

The filler applied to non-printing areas shall be either glue, collodion, shellac, lacquer, varnish or other approved filler. In each case the filler shall leave the screen open in areas thru which the paint must flow. Block-out stencils should not be employed for lettering less than 1/8 inch high.

3.3.2 Resist

These stencils shall employ either lithographer's tusche, japan colors or asphaltum to clog the pores of the screen. Cold glue may be applied over the stencil to avoid pinholes. The use of shellac or lacquer for this purpose is also permitted. No. 12 to No. 18 silk is satisfactory for these screens.

3.3.3 Paper

White sulphite paper, glassine paper, kraft paper, shellaced paper, lacquered paper, glue-paper, and vellum tracing have advantages but they are limited to quantities of 500 reproductions. No. 8 to 10 silk is satisfactory for screens using paper stencil.

3.3.4 Shellac or lacquer film

Stencils made of these materials must be cemented to the screening after the glassine backing paper is removed by lacquer thinner in the case of lacquer and by a hot iron in the case of shellac. No. 10 bolting silk cloth is adequate for these screens.

3.3.5 Cellophane or celluloid

These materials may be used for stencils but they must be cemented to the screen in a workmanlike manner and be cleanly and sharply delineated.

3.3.6 Photo-stencils

These shall be composed of gelatin, albumen, glue or polyvinyl alcohol-polyvinyl acetate rendered light sensitive by the addition of ammonium dichromate or potassium dichromate and applied to No. 13 or No. 16 silk bolting cloth (No. 16 cloth being used for fine detail).

3.4 Printing or Paint Mediums

3.4.1 Inks

3.4.1.1 Porous surfaces

Inks must comply with Specification TT-I-559B with the possible deviation that consistency may have to be altered.

3.4.1.2 Non-porous surfaces

Inks must comply with Specification TT-I-558B, with the possible deviation that consistency may have to be altered.

3.4.2 Oil paints

Paints sometimes called process paints, shall comply with Type I or Type II requirements of Specification TT-P-98 (1).

3.4.3 Dyes

When textiles are silk screened, the dye manufacturer's recommendations are to be followed.

3.4.4 Enamels

These materials must comply with Specification MIL-E-10242 and MIL-E-7729A with the possible exception that the gloss may be flattened.

3.4.5 Varnish

Whether it is used to reduce viscosity or to produce gloss on finished parts, varnish must not be brittle or embrittle the reproduction. See paragraph 6.1 of this specification.

3.4.6 Lacquer

Gloss or semi-gloss lacquers and lacquer pastes shall conform to Specifications TT-L-26 and MIL-L-52043 A (ORD) except with regard to consistency or viscosity. The percentage of volatiles may vary from the specified percentage to accomplish the desired consistency.

3.4.7 Vitreous enamels

When vitreous enamels are applied No. 4 to No. 8 screens shall be used. The paint manufacturers directions shall govern process. See paragraph 6.4 of this specification.

3.4.8 Thinners

3.4.8.1 Paint thinners

Varnish or turpentine may be used to thin the paste form of process paints. Turpentine flattens the gloss and robs the paint of some of its adhesive quality and should be used sparingly.

3.4.8.2 Lacquer thinner

Only the lacquer thinners recommended by the lacquer manufacturer should be used.

3.5 Equipment

3.5.1 Squeegee

These hand tools of appropriate dimensions for the job shall consist of a rubber strike or straight-edge held in a smooth finished handle made of seasoned wood or metal. The working edges of the rubber shall have a square edge and be of rubber possessing a durometer hardness of 40 to 60 (40, for soft grade; 50, for medium grade; and 60, for hard grade). Squeegees may also be made completely of soft, white pine, tapered at one end like a wedge.

3.5.2 Frames

These supports for the screening must be made of seasoned lumber or be of aluminum or steel. If they are of wooden construction, the corners must be reinforced by angle braces or by mortises or splines. No size limitations are imposed. The frame must permit the printing to be accomplished and must maintain the screening taut.

3.5.3 Miscellaneous equipment

Printing bases must be suitable and sufficient to accomplish the reproduction.

Counterbalances or hinges for removing the screen from flat work must function satisfactorily.

Registry of screens must be accomplished within 1/32 of an inch on multi-colored work by the choice of suitable guides, locators or line-up.

Racks for drying must allow for air circulation and be horizontal or vertical depending on the drying shrinkage characteristics of the printed base.

A light-box for tracing from originals must be of a adequate size and produce uniform, shadowless illumination.

Stencil cutting knives must be sharpened and honed to permit clean cutting of any stencil material.

3.6 Procedure

3.6.1 General

The characteristics of each reproduction will dictate the choice of stencil material or the type of screen material.

3.6.2 Block-out stencils

These stencils are to be made by placing the silk screen over the original and tracing the outlines with pencil or India ink. The screen is removed from the original and placed over a white surface to increase contrast and to ease the job of sealing the pores of the non-printing areas with tinted glue. When the glue has dried and the screen is satisfactory for printing, paint is poured on the screen and printing begun.

If shellac, lacquer or collodion is used instead of glue as a sealant, the silk screen is sized all over with glue (mixed in the ratio of 1 part of glue to 5 parts of water). When the sizing is dry, the screen is lain over the original and the design painted around the outline of the original. The screen is then removed from the original and the glue is removed by wiping the top side of the screen with a water moistened cloth. The screen must be held horizontally to prevent loosening the glue beneath the areas painted with sealant.

3.6.3 Resist stencils

These stencils are to be made by placing the screen over the original and tracing the design by pencil or India ink. The screen is then removed and its undersurface sized by use of a sponge moistened with a corn starch mixture of 1 tablespoon of ordinary corn starch to a glass of cold water. After drying for 15 minutes the original is copied with tusche and allowed to dry for 30 minutes. Two coats of cold glue are then scraped over the top surface of the tusche to seal pin holes. When glue is dried, a cloth saturated with turpentine, kerosene, naphtha, or benzene is rubbed on the underside of the silk screen. These solvents will melt the tusche and thereby remove the corn starch and tusche, but they leave the glue unaffected. The screen is dried by rubbing with dry cloths.

3.6.4 Film stencils

These stencils when made of lacquer or shellac film are to be traced as follows. The original drawing shall be placed on a flat surface, covered with a sheet of celluloid and then topped with the future film stencil. The corners are fastened with Scotch tape and the drawing or original duplicated by incising the film with a film stencil knife.

Lacquer stencils are cemented to the screen with lacquer thinner which is applied to the underneath side of the screen by a dampened cloth.

Shellac stencils are secured by applying a hot iron to the stencil which is protected by a sheet of paper.

The glassine paper backing is then carefully removed after the stencil has adhered to the screen.

3.6.5 Paper stencil

The handling of paper stencils is analogous to film stencils. The only difference would be with the use of opaque, kraft paper, in which case the operator or artist would sketch directly onto the paper. Since paper stencils are used for heavier coats of paint and yield an embossed appearing result, the paint used must be of thicker consistency. This heavier viscosity is utilized to cause the stencil to adhere to the screen. In some instances a small quantity of lacquer applied from the underneath side of the screen may be used to accomplish adhesion.

3.6.6 Cellophane and celluloid stencils

These stencils are handled analogously to film stencils. The cellophane because of its thinness is tacked with rubber cement to the original for tracing to prevent wrinkling. It is given a thin coat of shellac and allowed to dry before cutting. They are adhered to the silk screen by use of a hot iron in the same way as shellac stencils.

Celluloid stencils are very durable, but they are difficult to cut. They yield a heavy coat of paint. They may be cemented to the screening with the following preparation.

- | | |
|--------------------|-----------|
| ● Ethyl ether | - 5 parts |
| ● Acetone | -5 parts |
| ● Celluloid scraps | -2 parts |
| ● Camphor | -1 part |

3.6.7 Photo-stencils

Photo stencils may be prepared by coating the silk or bronze screening with light sensitized albumen, gelatin, glue, or polyvinyl alcohol-polyvinyl acetate. The gelatin coating is prepared as follows.

- | | |
|-----------------------------------|---------------------------------------|
| Nelson's, No. 1, gelatin | 1 ounce |
| Water | 10 ounce |
| Na_2CO_3 | 10 grains |
| $\text{K}_2\text{Cr}_2\text{O}_7$ | 96 grains (Approximately 2% solution) |

Into the top pan of an enameled or granite-ware double boiler (do not use metallic houseware) pour the water, add the gelatin and let it swell for 20 to 30 minutes. Next assemble the two portions of the double boiler to which water has been added to the pan in contact with the heating source. Heat the gelatin until it is dissolved but do not boil. Add the rest of the ingredients and stir with a glass rod. The render details of the future stencil more visible, a bit of aniline dye may be added.

Apply the hot gelatin to the screen with a soft, wide, clean brush making horizontal strokes on one side and vertical strokes on the other side of the screen. Allow the screen 30 minutes to dry before an electric fan. In the meantime discontinue heating the gelatin in the double boiler. Apply a second gelatin coat and allow it to dry. If the gelatin becomes lumpy, reheat the gelatin before applying the third coat. All operations must be done in a room dimly lighted with a 25 watt bulb provided the screen is held away from the light source. A candle or ruby lamp is the preferred illumination. The gelatin solution becomes fully light sensitive only when it has dried.

Transparent copy or originals are to be placed face up against the screen and exposed over a light box. Prior to exposure the top surface of the screen should be covered with black velvet, smoothed out, and weighted to bring the screen absolutely into close contact with the original. Then make the exposure. As a guide, tissue paper drawings exposed for 16 minutes to a 300 watt bulb give excellent results.

The light protected areas which were not hardened by light are dissolved in water at 105 to 110° F. As soon as the screen is wet, the lights may be turned on. Continue rinsing in the warm water until the open parts of the stencil are clear. The stencil is now dried by placing a lintless soft cloth in contact with each side and gently pressing. This technique removes most of the water. The stencil is finally dried by an electric fan.

To remove the stencil from the screen after use, employ the following solution:

NH ₄ OH(20%)	4 ounces
NaOH	1 pound
Water	2 gallons

3.6.7.1 Other photo-stencils

Super-Duro, Photoetch Photox and Ektagraph films mounted on No. 16 or No. 18 silk bolting cloth are proven stencils. The procedure requires the use of a sensitizing solution applied liberally by brush and forced air dried. It is then exposed in contact with a positive or transparent original, rinsed in warm water at a maximum of 120°F, rinsed in cold water and finally cemented to the bottom side of the screen by laying the screen over the film and patting with dry newspapers until all moisture is removed. Use a fan to dry the film thoroughly.

When the film is dried, the backing sheet is removed. The screen is now ready for printing.

The screen is cleaned after printing by washing with hot water mixed with 5 percent ammonia water. Rinse the screen finally with cold water.

3.7 Printing Method

After the screen frame is positioned over the material to be printed, sufficient ink or paint for 20 reproductions is poured onto the screen. The squeegee is moved across the screen from end to end, and the operator makes certain that sufficient paint is ahead of the squeegee to cover the area to be printed. The screen frame is then removed and the material that was printed removed to a drying rack.

If fountain brushes or other applicators are used, no ink or paint is poured onto the screen and no squeegee is necessary.

If a spray gun is used, the stencil must be cleaned in suitable thinner immediately after finishing the lot. The thinner must not attack the stencil.

4. MANUFACTURING CONTROL PROVISIONS

4.1 General

The content of this specification is meant to be descriptive instead of restrictive. This is necessary because the characteristics of the job may require deviations to achieve the quality obviously required by drawings and purchasing agreements.

5. PREPARATION FOR DELIVERY

5.1 General

Not applicable.

6. NOTES

6.1 Intended Use

This manufacturing specification, developed by the Process Engineering Laboratory of the George C. Marshall Space Flight Center, is intended for use in the manufacture of components for space flight vehicles.

6.2 Long and Short Varnishes

When reproductions are coated with varnish to preserve them, a glossy or semi-glossy varnish is desired which has sufficient flexibility to resist cracking. When oil must be added to improve consistency, a long oil should be used.

The term, "long", also applies to colors ground in dammar, resin, various copals; i. e. Kauri, Manila, or synthetic resins.

The term, "short," applies to colors that dry flat and are produced by adding oleum spirits.

6.3 Oil Paints

Sometimes a transparent base made of aluminum stearate or aluminum palmitate is added for the following reasons.

- Thicken colors.
- Make oil colors shorter, i. e. has too much oil. In this condition the color blurs and spreads on the print.
- Make colors transparent.

6.4 Printed Circuits

Silk screens are to be prepared photographically from positives drawn on Mylar plastic, 0.0075 inch thick, using acetate base inks. A photographic negative is produced and a Kodak-Photo-Resist, treated copper-clad board is exposed through the negative and subsequently developed by trichloroethylene. The unprotected copper is leached away by a ferric chloride solution. The remaining copper constitutes the printed circuit.

6.5 Plated Circuits

Photo-stencils produced from a photographic negative can be used to apply Kodak-Photo-Resist or No. 205 NAZ Dar plating resist onto copper-clad boards whenever the printed circuit is to be plated with precious metals. After trichloroethylene dissolves the Kodak-Photo-Resist the ferric chloride solution etches away the copper, but it leaves the precious metal protected copper undisturbed.

6.6 Fused Printed Circuits

Ceramic articles are sprayed with copper oxide containing frit thru silk screens. The ceramic article is fused to embed the frit. The copper oxide is subsequently reduced in a hydrogen atmosphere to metallic copper which forms the conductor.

6.7 Wire Screens

These screens are very stable. They ease the difficulty of achieving registry on multi-colored work. They are very easily used on direct emulsion work. On exceedingly fine detail they have proven to be the best. They permit more paint to pass through uniform apertures. Sodium hydroxide used for stripping stencils has no effect on the screen. They are capable of lasting an untold number of stencils.

Though wire isn't easy to stretch and dents are almost impossible to remove, the problem is eased by using a circular frame.

NOTICE

When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government

procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Custodian

Process Engineering Laboratory
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Preparing Activity

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George C. Marshall Space Flight Center

M-ME-MPROC-150.9

MANUFACTURING PROCESS FOR
SURFACE TREATMENT OF WELD AREAS OF THE
S-IC BULKHEAD TEST FUEL TANK

1. SCOPE

1.1 Scope

This document covers the approved interim procedure of the Process Engineering Laboratory for the surface treatment of weld areas of the S-IC bulkhead test fuel tank following welding of the bulkhead.

1.2 Applicability

The surface treatment procedures specified in this manufacturing process are applicable to 2219 aluminum alloy of the following heat treatment conditions: T-81, T-87, and T-852.

2. APPLICABLE DOCUMENTS

2.1 Governmental

The following documents, of the issue in effect on the date of use of this process, form a part of this process.

SPECIFICATIONS

George C. Marshall Space Flight Center

MS 120.2A	Deoxidation of Aluminum Alloys
MS 150.3	Application of Conversion Coatings to Aluminum Alloys
M-ME-MPROC 150.7	Surface Treatment of Skin and Bulkhead Material for the S-IC Vehicle
M-ME-MPROC 200.1	Welding of Bulkhead Gores for the S-IC Vehicle
M-ME-MPROC-200.2	Welding of Fittings into the Bulkhead for the S-IC Vehicle

Military

MIL-C-5541

Chemical Films for Aluminum and Aluminum Alloys

Federal

0-T-634

Trichloroethylene, technical

0-E-760

Ethyl Alcohol (Ethanol); Denatured Alcohol; and Proprietary Solvent

2.2 Other Publication

The following document, of the issue in effect on the date of issuance of this specification, forms a part of this specification.

#14-2 A1 Coat

Operating Data for Iridite #14-2
A1 - Coat for Aluminum and Aluminum Alloys

(Copies of this publication may be obtained from Allied Research Products, Inc., 4404 E. Monument St., Baltimore 5, Maryland)

3. REQUIREMENTS

3.1 Equipment

3.1.1 Spray Equipment

A stainless steel spray gun with a cup capacity of approximately 1 quart shall be used when spray applying the conversion coating solution. The pressure pot shall be constructed of stainless steel and have an approximate capacity of 2 gallons.

3.1.2 Paint Brush

Disposable brushes shall be used when brush applying the conversion coating solution.

3.2 Materials

3.2.1 Cleaning Solvent

Ethyl alcohol conforming to Specification 0-E-760b or trichloroethylene conforming to Specification 0 T-634 shall be used.

3.2.2 Conversion Coating Solution

Iridite 14-2 A1 - coat at a concentration of 4 to 8 ounces per gallon of aqueous solution with a pH between 0.4 and 1.1 at ambient temperature shall be used. Adjust pH with nitric acid when conversion coating solution pH rises above 1.1.

3.2.3 Thickener for Conversion Coating Solution

Cab-O-Sil, a silica based thickener, shall be added to the conversion coating solution. The concentration shall be 5 to 7 ounces of Cab-O-Sil per gallon of conversion coating solution. Cab-O-Sil is available from Cabot Corporation, Minerals and Chemicals Division, 125 High Street, Boston, 10, Massachusetts.

3.2.4 Demineralized Water

Filtered water, possessing a minimum electrical resistivity of 50 000 ohms, shall be used for rinsing and for make-up of the conversion coating solution.

3.3 Procedure

3.3.1 Mechanical Cleaning

Weld areas and areas adjacent to welds not covered by a conversion coating shall be mechanically cleaned by hand or grinding operation using non-electrolytic material (aluminum wool or aluminum oxide abrasive).

3.3.2 Solvent Cleaning

Solvent clean with ethyl alcohol conforming to Specification 0-E-760 or trichloroethylene conforming to Specification 0-T-634 (Type I or II). Allow surface to dry.

3.3.3 Conversion Coating

Brush or spray the conversion coating solution (4 to 8 ounces Iridite 14 to 2 plus 5 to 7 ounces Cab-O-Sil per gallon of solution) over the entire uncoated weld area.

3.3.4 Rinsing

Sponge or wipe off excess conversion coating solution from the weld area with demineralized water. Wipe all surrounding area with clean dry cloth to remove all moisture. Allow to dry for a minimum of 24 hours at ambient temperature.

4. MANUFACTURING CONTROL PROVISIONS

4.1 Visual Examination

Conversion coatings usually exhibit an iridescent color ranging from light golden to brown. The coating shall be continuous, smooth, adherent, powder-free, and uniform in appearance.

4.2 pH Determination

The pH of the conversion coating solution shall be maintained between 0.4 and 1.1. pH determinations shall be made electrometrically using a potentiometer with a glass electrode.

5. NOTES

5.1 This manufacturing process has been coordinated within the Process Engineering Laboratory and is intended for use in the surface treatment of weld areas of the S-IC bulkhead test fuel tank. Final verification of this procedure will be established during the production of the first test vehicles.

5.2 Any deviation from this basic procedure will require concurrence from Process Engineering Laboratory. An emergency concurrence may be made by telephone, but it must be confirmed in writing.

CAUTION

String, wood, paint, or other organic material shall not be placed in the conversion coating solution.

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

MANUFACTURING PROCESS FOR THE APPLICATION OF
DYE PENETRANT TO TANK SURFACES
(NON-LOX WETTED) FOR S-IC VEHICLE

1. SCOPE

1.1 Scope

This manufacturing process covers the approved Process Engineering Laboratory requirements for the pre-cleaning of parts prior to the application of dye penetrants, the application of the dye penetrant, and the subsequent rinsing operations.

1.2 Applicability

The process applies to all S-IC fuel tank surfaces, namely all skin segments and all bulkhead gores.

2. APPLICABLE DOCUMENTS

2.1 Governmental

The following documents, of the issue in effect on the date of use of this process, form a part of this process.

SPECIFICATIONS

George C. Marshall Space Flight Center

MS 150.0 Vapor Degreasing of Metallic Surfaces

MS 150.3 Application of Conversion Coatings to Aluminum Alloys

M-ME-MPROC 150.7 Surface Treatment of Skin and Bulkhead Material for the S-IC Vehicle

Federal

0-T-634 Trichloroethylene, technical

Military

MIL-STD-271

Nondestructive Testing Requirements for Metals

MIL-I-6866

Inspection, Penetrant Method of

2.2 Other Publications

The following documents form a part of this process. Unless otherwise indicated, the issue in effect on the date of issuance of this process shall apply.

Allied Research Products, Inc.

No. 14-2 Al-Coat Bulletin Operating Data for Iridite No. 14-2 Al-Coat for Aluminum and Aluminum Alloys

(Copies of this publication may be obtained from Allied Research Products, Inc., 4404 E. Monument St., Baltimore 5, Maryland.)

Turco Products, Inc.

Bulletin No. 78

Turco 4215 (Non-Silicated Alkaline Spray Washing and Hot Tank Compound for Use on Aluminum)

Bulletin No. 84

Turco 2897 Redstone (Smut Go)

(Copies of these publications may be obtained from Turco Products, Inc., P. O. Box 1055, Wilmington, California.)

3. REQUIREMENTS

3.1 General

Not applicable.

3.2 Materials

3.2.1 Zyglo-ZL-22 Dye Penetrant

All Zyglo products listed in this process are available from Magnaflux Corporation, Atlanta, Georgia. This material shall be used at full strength.

3.2.2 Zyglo ZE-3 Emulsifier

This material shall be used at full strength.

3.2.3 Zyglo ZP-5 Developer

The developer solution shall consist of 1 pound of Zyglo ZP-5 Developer per gallon of water.

3.2.4 Turco 4215 Alkaline Cleaner

The alkaline cleaning solution shall contain 6 to 12 ounces Turco 4215 per gallon of water. The temperature shall be 140 to 180°F and the pH shall be 2 to 4.

3.2.5 Turco 2897 Redstone (Smut Go)

The deoxidizer shall contain 8 to 16 ounces of Turco 2897 Redstone per gallon of water. The solution shall be used at ambient temperature and the pH shall be 2 to 4.

3.2.6 Iridite 14-2

The conversion coating solution shall contain 1.5 to 2.25 ounces Iridite 14-2 per gallon of aqueous solution with a pH between 1.2 and 1.7 at ambient temperature.

3.2.7 Wyandotte mil etch

The etch solution shall contain not more than 12 ounces per gallon of Wyandotte Mil Etch.

3.2.8 Paper

Oil free paper shall be used in the packaging operation.

3.3 Equipment

The spray equipment listed below is available from the Magnaflux Corp., Atlanta, Georgia

3.3.1 Dye penetrant spray

The spray equipment necessary for the application of dye penetrant solution to the parts is as listed.

- One - D-5500 5-gallon capacity tank.
- One - 2716 spray gun.
- One - 25 foot length of 3063 air hose.
- One - 25 foot length of 4645 fluid hose.
- One - SX2003 air pressure regulator.
- One - SX2004 air gauge.

3.3.2 Emulsifier spray

The spray equipment necessary for the application of the emulsifier solution to the parts is as listed.

- One - D-5500 5-gallon capacity tank.
- One - 2702 spray gun with a #66 fluid nozzle.
- One - 25 foot length of 3063 air hose.
- One - 25 foot length of 4645 fluid hose.
- One - SX2003 air pressure regulator.
- One - SX2004 air gauge.

3.3.3 Wet developer spray

The equipment necessary for the application of the wet developer solution shall be the same type as the equipment outlined in paragraphs 3.3.1.1 through 3.3.1.6. Do not use the same equipment that has been used for dye penetrant.

3.3.4 Infrared heating lamps

The standard 250 watt bulbs shall be used.

3.4.1.1 Solvent cleaning

Vapor degrease by lowering into trichloroethylene vapors at 180°F (± 3) at approximately 11 feet per minute. Allow the vapors to condense and flow from the skin or bulkhead material until the component reaches the temperature of the vapor. Remove the component at a rate not greater than 11 feet per minute. An approved equivalent method may be used for this step.

3.4.1.2 Alkaline cleaning

Alkaline clean with Turco 4215 by dip immersion for 20 minutes at 140 to 180°F. The solution shall have a pH of 8 to 10. An approved equivalent method may be used for this step.

3.4.1.3 Rinse

Rinse with hot water (160 to 180°F) until pH of aluminum surface is between 6 and 8. If surface is not water-break free, repeat paragraph 3.4.1.1 and 3.4.1.2.

3.4.1.4 Etch cleaning

Etch from .0004 to .001 inch from the part using Wyandotte Mil Etch solution at ambient temperature. An approved equivalent method may be used for this step.

3.4.1.5 Rinse

Rinse by spray and/or immersion with hot water (160 to 180°F).

3.4.1.6 Deoxidation

Deoxidize with Turco 2897 Redstone (Smut-Go) for 30 minutes at a concentration of 8 to 16 ounces per gallon of water. The pH of the solution will be between 2 to 4. This operation will be performed at ambient temperature. An approved equivalent may be used for this step.

3.4.1.7 Rinse

Rinse with ambient temperature water until the pH of the aluminum surface is between 6 and 8.

3.4.1.8 Drying

Dry by heating with hot air blast until completely dry. Further processing shall be delayed until the part has cooled to 100°F or below.

3.4.2 Process II, Dye Penetrant Application

3.4.2.1 Dye Penetrant Application

Before application of dye penetrant, the part must be completely dry and the surface temperature shall be 50 to 100°F. Apply Zyglo ZL-22 by spraying in a manner to insure complete coverage of the part. The penetrant shall remain on the surface for 20 minutes.

3.4.2.2 Emulsifier Application

The emulsifier shall be sprayed on in a manner to insure complete even coverage, beginning at the bottom and continuing to the top of the part. The emulsifier shall be applied to only one side of the part at a time and allowed to remain on the part a minimum of 2 minutes and a maximum of 2 minutes 30 seconds. The spray gun to be used will conform to the requirements stated in paragraphs 3.3.2.2.

3.4.2.3 Rinse

While using a source of warm (90 to 100°F) water, tap or demineralized, begin rinsing the solutions from the part. The rinsing operation shall be started at the bottom of the part and rinsing continued until 5 minutes. The part shall be rinsed one side at a time and particular care shall be taken to avoid wetting the opposite side.

3.4.2.4 Developer Application

Using the spray equipment specified in paragraph 3.3.3 apply a uniform coat of ZP-5 developer that has been mixed in accordance with the specification outlined in paragraph 3.2.3. The nozzle of the spray gun used in this operation must be completely cleaned before use. Spray a uniform coating of this material over the entire part.

3.4.2.5 Drying

Allow the part to dry at room temperature for a minimum time of 30 minutes and possibly up to 4 hours until completely dry.

3.4.2.6 Inspection

An inspection will be made of each treated part by the Quality Assurance Division. If the part is approved for production proceed to Process III, paragraph 3.4.3, Removal of Dye Penetrant.

3.4.2.7 Rejected Parts

An evaluation will be made of all rejected parts to determine whether rework is required. If rework is required, then the part shall be reprocessed as outlined in paragraphs 3.4.1.2 through 3.4.2.5.

3.4.3 Process III, Removal of Dye Penetrant

3.4.3.1 Rinse

Using hot water (130-140°F) rinse the part by spraying and/or dip immersion for at least 30 minutes and let dry.

3.4.3.2 Solvent Cleaning

Vapor degrease by lowering into trichloroethylene vapors at 188°F (±3) at approximately eleven feet per minute. Allow the vapors to condense and flow from the skin or bulkhead material until the component reaches the temperature of the vapor. Remove the part at a rate not greater than eleven feet per minute. Allow the part to dry at room temperature. As approved equivalent method may be used for this step.

3.4.3.3 Alkaline Cleaning

Alkaline clean with Turco 4215 by dip immersion for 20 minutes at 140 to 180°F. The concentration of cleaner shall be 6 to 12 ounces per gallon of water and at a pH of 8 to 10. An approved equivalent method may be used for this step.

3.4.3.4 Rinse

Rinse with deionized water (160 to 180°F) until pH of aluminum surface is between 6 and 8. If surface is not water-break free, repeat paragraphs 3.4.1.1 and 3.4.1.2.

3.4.3.5 Deoxidation

Deoxidize with Turco 2897 Redstone (Smut-Go) for 30 minutes at a concentration of 8 to 16 ounces per gallon of water at ambient temperature and a pH of 2 to 4. An approved equivalent method may be used for this step.

3.4.3.6 Rinse

Rinse with ambient temperature demineralized water until pH of aluminum surface is between 6 and 8. Let the part stand until dry.

3.4.3.7 Drying

Allow to remain at room temperature until dry.

3.4.3.8 Inspection

An inspection will be made of each part by the Quality Assurance Division to insure that all traces of the dye penetrant have been removed.

3.4.4 Process IV, Re-Conversion Coating

3.4.4.1 Re-Conversion Coating

Conversion coat with Iridite 14-2 by dip immersion for 1-1/2 to 3 minutes at ambient temperature. The bath shall contain 1.5 to 2.25 ounces of Iridite 14-2 per gallon of water with a pH between 1.2 and 1.7. Alodine 1200 may be used in place of Iridite 14-2.

3.4.4.2 Rinse

Rinse with ambient temperature demineralized water until pH of aluminum surface is between 6 and 8.

3.4.4.3 Drying

Allow the part to air-dry at room temperature.

3.4.5 Packaging

The part will be wrapped with oil-free paper and returned to storage.

4. QUALITY ASSURANCE

4.1 Inspection Tests

4.1.1 After Developer

Quality Assurance Division will inspect parts for defects by the use of black light.

4.1.2 After removal of dye penetrant and developer

Quality Assurance Division will inspect with black light to assure complete removal of all traces of dye penetrant.

Toxicity - The Zyglo ZL-22 Dye Penetrant, Emulsifier ZE-3 and Developer ZP-5 are non-toxic, therefore, special masks and other equipment are not required.

Emulsifier ZE-3 and Zyglo ZL-22 Dye Penetrant both have a flash point of 210°F when tested in a TAG closed cup. This temperature must be avoided where vapors of these materials are present.

Developer ZP-5 is non-flammable.

Any deviation from this basic procedure will require written concurrence from Process Engineering Laboratory. An emergency concurrence may be made by telephone, but it must be confirmed in writing.
in writing.

Preparing Activity

Process Engineering Laboratory
George C. Marshall Space Flight Center

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